

# ANNUAL REPORT

1959-60



NATIONAL METALLURGICAL LABORATORY

COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH

NATIONAL  
METALLURGICAL LABORATORY

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JAMSHEDPUR, INDIA



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## FOREWORD

I HAVE great pleasure in presenting this report on the working of the National Metallurgical Laboratory during the year 1959-60.

The Laboratory, now in its ninth year, has well-nigh completed the expansion envisaged under the Second Five Year Plan and is preparing itself for shouldering larger responsibilities in the next Plan period. Its plans for expansion, completed or projected, are closely allied to problems of the rapidly expanding mineral and metal industries.

Greater emphasis is being progressively laid in the Laboratory's work on the industrial implementation of research through the agency of pilot plant trials and investigations. It is in this sphere of the Laboratory's activities that the greatest expansion has taken place during the Second Plan. This is a healthy development because pilot plants help to speed up the translation of the latest technological concepts and innovations into acceptable industrial practice. Significantly, therefore, the annual Symposium held in February 1960 was devoted to the discussion of the role "Pilot Plants in Metallurgical Research and Development". A new technological block, housing four new pilot plants, was inaugurated during the Symposium. Particularly gratifying is the fact that these new pilot plants were designed and fabricated in the Laboratory's Instrument and Machine Workshops, saving the country's considerable foreign exchange. Apart from pilot plants, these workshops have successfully fabricated many other items of costly scientific equipment.

The largest pilot plant, of course, is the 15-ton per day low-shaft furnace set up in January 1959. Apart from the usual teething troubles in operating a new plant of this size, several basic modifications had to be made by the Laboratory to the component units. Over a thousand tons of foundry grade pig iron of suitable quality have, however, been produced. The current programme of work relates to the smelting of iron ores from Bihar and Orissa with non-coking coals from the Raniganj and Dishergarh coalfields. This is to be followed up by smelting trials on iron ores from Chanda district and non-coking coals from Ballarpur, Kampti and Wardha valley in Maharashtra State. Later, Salem magnetite ores and Neyveli lignites will be used for experiments.

Another important line of research is the effort to develop a rationalized series of alloy steels based on indigenous elements. The Laboratory is also working on basic lined, side air-blown converters of varying capacities for making steel from medium phosphoric indigenous pig irons. Thus, this Laboratory is developing as a central institution for operational and development research in the iron and steel industry.

In respect of austenitic nickel-free stainless steel, research has been directed towards the problems of industrial scale production, particularly the rolling down of tonnage ingots into commercial gauges of hot-rolled and pickled sheets. These sheets were subjected to various tests, including the assessment of the

deepdrawing properties, both in cold and hot-rolled conditions for utensils and other applications. It is a matter of satisfaction that all raw materials used in these tests were processed in the Laboratory right from the stage of ores.

Another approach of importance is the effort to substitute nickel by other indigenously available alloy elements for the production of heating elements and coinage alloys. At the request of the Defence Ministry, the Laboratory has been working on the production and supply of low-carbon ferro-chrome, in which considerable success has been achieved. Likewise, magnesium powder of high purity has been successfully made on a laboratory scale conforming to the rigid specifications of the Ministry. A Pilot Plant for the production of magnesium from dolomite is now being established.

Beneficiation of low-grade manganese ores is a subject which has been under continuous study. Ore samples from various States were investigated and the feasibility of upgrading established. The complexity of these investigations has been made public in a monograph entitled "Beneficiation of Low-grade Indian Manganese Ores". Another monograph on "Foundry Moulding Sands of India" is under print.

During the year, the Laboratory took out several patents. Practical demonstrations were held to acquaint interested manufacturers and engineers with the know-how of four patented processes. These were released free of royalty-premia for the benefit of small-scale industries. This apart, it may be noted that some processes developed here are now being commercially exploited on payment of royalties. Shortly expected to be added to this list is the process for the electrolytic production of manganese and manganese dioxide. To extend the benefit of research and development work done in the Laboratory, the Liaison and Information Service has been enlarged and considerable importance is attached to providing technical aid to small and medium-scale industries.

I take this opportunity to recall our gratitude to the U.K. Government for making a gift of equipment worth over a million rupees under the Colombo Plan. The formal presentation was made during the February Symposium by the U.K. Trade Commissioner to Prof. M. S. Thacker, Director-General, Scientific & Industrial Research.

In the present stage of the Laboratory's development, priority is naturally given to operational and development work, but fundamental research has not been ignored. Several important studies are in progress which are illustrated in the appropriate section of the report.

The Council of Scientific & Industrial Research are proving their usefulness in various fields of National Development and I am happy to note that the National Metallurgical Laboratory is holding a prominent place therein. I should like to express our deep appreciation of the hard work and devotion to duty of Dr. B. R. Nijhawan, Director of the Laboratory, and his colleagues. They have done commendable work in many fields and much of the success of the Laboratory's work is undoubtedly due to the organizational ability of Dr. Nijhawan.

J. J. GHANDY

*Chairman*

*Executive Council of the*

*National Metallurgical Laboratory*

Jamshedpur  
May 1, 1960



## INTRODUCTION

During the year under review, the National Metallurgical Laboratory has not only maintained intensive and ceaseless activity in multitude directions but also has notably stepped up the tempo in the field of implementation of pilot plant projects. The new Pilot Plant Technological Bay, built at a cost of about Rs. 4 lakhs in the record time of just over a year, was declared open and four important pilot plants, such as the aluminizing of steel wire and sheet, mineral and thermal beneficiation of low-grade Indian ores, particularly manganese and chrome, production of electrolytic manganese and manganese dioxide, development of indigenous refractories for steel and metallurgical industries, are located in this Pilot Plant Technological Bay. The total value of these pilot plants will be in the neighbourhood of Rs. 25 lakhs. The designing of the different sections of these pilot plants and fabrication of most of the components needed were done in the National Metallurgical Laboratory, which is a matter of considerable gratification for us. Another notable field in which good progress has been made is the handing over of our research processes on to the industry on payment of lump-sum premia and royalties. The electro-metallurgical processes to produce electrolytic manganese and manganese dioxide from low-grade Indian manganese ores are now on the point of passing on to the Indian industry, while the process entitled "Improvement in or relating to mullite refractories from kyanite" has already been handed over to industry. Other processes are awaiting industrial scale implementation and it is hoped to maintain the rate of such industrial scale utilization of the National Metallurgical Laboratory's patented and non-patented processes — in

the latter category the process entitled "Liquid Gold" is now ready for handing over to several industries on non-exclusive basis on payment of handsome premia and production royalties.

The continuous operation on round-the-clock basis of the Low-shaft Furnace Pilot Plant has yielded valuable and encouraging results in the utilization of non-coking coals for the production of foundry grade pig iron. Over a thousand tons of foundry grade pig iron worth several lakhs of rupees are now ready for sale and represent the first fruits in India of smelting iron ore fines with non-metallurgical fuels that cannot be used in the conventional iron blast furnace technology. The operation of this Low-shaft Furnace Pilot Plant has given the staff considerable operational experience of the constituent briquetting plant, low-shaft furnace proper and the gas-cleaning plant on an integrated basis and in their efficient maintenance besides confidently tackling operational hazards met with. For further investigations, raw materials from the Government of Maharashtra on free basis have now been received and are awaiting large-scale trials. The Government of the East Punjab likewise has come forward to give us raw materials also on free basis for large-scale trials on their iron ore deposits in the Narnaul district of the East Punjab. These pilot plant scale trials will be followed by similar trials on raw materials from other parts of India, particularly the smelting of Salem magnetite ores with high temperature carbonized lignite from Neyveli. It is in these and related industrial fields that the usefulness of the National Metallurgical Laboratory is today being not only recognized by the industry both in the public and private sectors but also freely availed of by them.



Different industrial organizations, Government Departments and Institutions have continued to call heavily upon the National Metallurgical Laboratory for technical advice, *ad hoc* investigations, survey of industrial projects and initiation of undertaking long-term research projects. An active Electronic Section, which was set up earlier, is now rendering useful service not only in the design and fabrication of ancillary electronic gadgets and instruments but also in their repair and maintenance of imported equipment. The Design Section, Mechanical and Electrical Workshops and other service units have continued to perform useful service of considerable value in the research and development projects under way at the National Metallurgical Laboratory. Whilst relevant details of the research projects under way are given in the following pages, reference may be made to the valuable progress made on research and development work on industrial scale production of chromium-manganese-nitrogen austenitic nickel-free stainless steel, study of its ageing, assessment of physical characteristics at elevated and sub-zero temperatures, electro-polishing and its potentiality as a high temperature creep-resistant material. Notable success has also been achieved on the development of nickel-free heating elements based totally on indigenous alloying elements, bimetals research and nickel-free coinage alloys and nickel-free manganese-based brasses; each of these important projects for the development of substitute alloys is progressing most favourably. Development work on mineral beneficiation and ore-dressing projects has continued to yield valuable results to the industry. The development and production scale work on different types of ferro-alloys, urgently needed by ordnance establishments, from indigenous materials made considerable progress as also the production of magnesium from indigenous dolomite prepared to rigid specifications laid down by the T.D.E. Establishment of the Ministry of Defence. Pilot plant scale work on the

production of magnesium — a highly important project — is now awaiting due sanction and subsequent establishment. Researches in physical metallurgy fields related to the fundamentals of physics of metals have made valuable progress such as studies on the transformation characteristics of austenite and detailed factors governing retained austenite. In the applied field also related work on magnetic alloys has been highly satisfactory. Progress in the fabrication of scientific equipment has been noteworthy and apparatus for the determination of thermal conductivity of different types of refractories, apparatus for Accelerated Life Test of Heating Elements as per A.S.T.M. specification, Hot Hardness Tester to determine hardness of metals at Elevated Temperature, Thermal Balance to determine the oxidation-resistance of stainless steel, besides other valuable equipment, have been fabricated and are in constant use. It goes without saying that foreign exchange worth several lakhs has thus been saved in the fabrication of laboratory scale apparatus and pilot plant scale equipment. Work in other directions has also been steady and highly rewarding. Liaison, information and publication work has considerably increased and is being efficiently handled. The publication *NML Technical Journal* continues to draw recognition from far and wide. The proceedings of the Symposium on "Iron and Steel Industry in India", held earlier in February 1959, has been published in the record time of a few months containing 45 authoritative papers and the records of discussion in over 500 pages and this publication has received wide appreciation in many overseas countries and at home. The monograph on "Beneficiation of Low-grade Manganese Ores of India" has also been published during the year under review and has also received wide appreciation at home and abroad. Likewise, the monograph on "Foundry Moulding Sands and Bonding Clays in India" containing a comprehensive account of related research and development work

undertaken at the National Metallurgical Laboratory is now in the final stages of publication. The last Symposium on "Pilot Plants in Metallurgical Research and Development" was a notable success; not only was it international in character in respect of delegates participating from all parts of the world, but also from the point of view of having attracted valuable technical and research papers from those distinguished in respective metallurgical fields at home and in different parts of the world. During the year under review, the magnificent gift of equipment worth Rs. 1 million from the British Government under the Colombo Plan was handed over by the United Kingdom Trade Commissioner to the National Metallurgical Laboratory.

During the year under review, the following patents have been filed:

1. 67871: A method for manufacture of porous bronze bearing — filed on 1-6-1959
2. 68171: Compositions and method of making welding flux — filed on 26-6-1959
3. 68174: Refractory compositions containing non-refractory chrome ore and refractory products made therefrom — filed on 26-6-59
4. 68401: Improvement in or relating to the manufacture of bricks or blocks out of ceramic mixes — filed on 15-7-59

Patents for the following processes have been accepted:

1. 62352: Refractory compositions comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation — accepted on 8-8-1959
2. 62938: A process to produce dense carbon aggregates from carbonaceous materials of varied volatile contents — accepted on 15-7-1959
3. 63904: A process for recovering zirconium dioxide from zircon — accepted on 16-9-1959
4. 65231: An improved method for the production of chromium-manganese alloys by alumino-thermic reaction — accepted on 6-8-1959

5. 65556: A process for electrolytic production of iron-chromium alloys from chromite ore — accepted on 6-1-1960

The following patents were sealed during the year:

1. 61978 (cognated with 61979 & 61980): New stainless steels and methods of preparing them — sealed on 9-9-1959
2. 61981: A process for making completely stabilized dolomite refractories — sealed on 8-7-1959

Non-technical notes on the following patented processes were prepared and circulated to interested industrial parties:

1. An improved method for the production of titanium-tetrachloride from ilmenite — Patent No. 58244
2. A process for the production of chemically bonded metal clad or unclad basic refractories — Patent No. 65610
3. Refractory compositions containing non-refractory chrome ore and refractory products made therefrom — Patent No. 68174
4. Liquid Gold (non-patented method).

The following patented processes were released to the industry on lump-sum premium and royalty:

1. Refractory compositions comprising graphite and silicon carbide — Patent No. 58869
2. Refractory compositions comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation — Patent No. 62352
3. Improvement in or relating to mullite refractories from kyanite — Patent No. 58533

Collaborative research has continued to make good progress with other scientific organizations in the country including the Metals Research Committee, Indian Standards Institution, Ministry of Defence, Railways, etc.

A brief résumé of the highlights of important problems under progress is given in the following pages.

# RESEARCH PROJECTS

## 1.0 Nickel-free Austenitic Chromium-Nitrogen-Manganese-Copper Stainless Steel

India possesses no reserves of nickel and imports at the expense of considerable foreign exchange heavy tonnages of nickel-bearing austenitic stainless steel, chiefly in the form of sheets for fabrication into utensils and chemical uses. Research and development work conducted at the National Metallurgical Laboratory have resulted in the formulation of processes for the production of nickel-free austenitic stainless steel from entirely indigenous raw materials. Comprehensive investigations on the determination of mechanical, physical and corrosion-resistance properties of the stainless steel in various media were conducted and it was found to possess excellent properties required for fabrication of utensils and other deep-drawn products for certain specific applications. Industrial scale production of chromium-

manganese-nitrogen austenitic steel was successfully carried out in direct arc-melting furnace and further work was directed towards the rolling down of the tonnage scale ingots into 18 to 22 gauge hot-rolled and pickled sheets. Complete mechanical and corrosion-resistance tests of the sheets were performed and deep-drawing properties both in the cold and hot-rolled condition were evaluated. Further work was extended on determining the hot-working properties of Cr-Mn-N stainless steel with the addition of increasing amounts of copper and certain other alloying elements. The effect of higher manganese contents on working properties of the steel was also studied.

### 1.1 Magnetic and Electrical Properties

Magnetic and electrical properties of the nickel-free austenitic stainless steel were also determined. Their average values are furnished below:

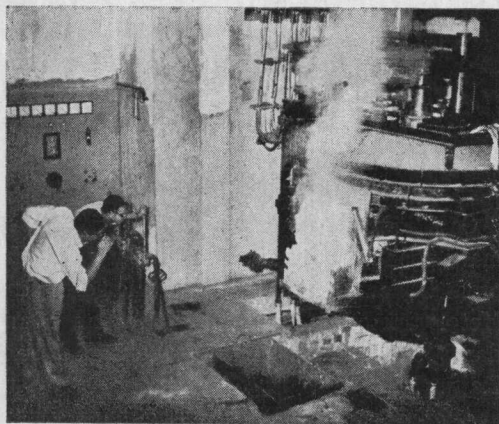
Electrical resistivity — 74.69 micro-ohm/cm.

Saturation induction — Less than 2500 gauss for magnetizing field of 2340 oersteds

Average permeability — 1.05

A few heats were made with pure chromium metal to determine the relative austenitizing behaviour of carbon and nitrogen at exceedingly low carbon contents. With a view to improve the surface condition of the ingots, the effects of capping on the ingot structure were studied by examining a series of sectioned 60 lb. ingots.

Intergranular corrosion tests were performed on Cr-Mn-N stainless steel following the



ONE-TON ELECTRIC ARC FURNACE INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY



A.S.T.M. method. Sub-zero impact tests were also made and the transition temperature ranges of the stainless steels were evaluated. A thermal balance has been designed and fabricated at the Laboratory to evaluate the oxidation-resistance of Cr-Mn-N stainless steel and tests were performed at 400°, 500°, 600° and 700°C. in still air. Physical characteristics at elevated temperatures including creep-resistance properties were also investigated. Experiments were also conducted to study the ageing characteristics of nickel-free Cr-Mn-N austenitic stainless steels at elevated temperatures. The specimens were taken from hot-rolled and solution-treated sheets and were subjected to different ageing temperatures for extended periods. After ageing, X-ray diffraction studies, metallographic examination, corrosion and hardness tests were carried out, besides physical properties and related characteristics at elevated and sub-zero temperatures.

## 1.2 Dilatometric Studies

To study the transformation temperatures and other thermal characteristics, heating and cooling curves were plotted for different ranges of nickel-free Cr-Mn-N stainless steels from several heats. The coefficient of thermal expansion of various ranges within the limits of experiment was also assessed.

## 1.3 Electrolytic Polishing of Stainless Steel

Investigations were conducted on sulphuric acid glycerine electropolishing baths for their suitability for polishing of various types of categories of stainless steels including chromium-manganese-nitrogen austenitic stainless steels, 18:8 Cr-Ni stainless steels and ferritic (17 per cent Cr) stainless steels. Baths with different compositions were investigated under various conditions of bath temperatures, current density, cell voltage and time of treatment to find the optimum

conditions of polishing. The investigations have shown that both at room temperature and higher temperatures, a minimum concentration of 30 per cent by volume of sulphuric acid is necessary for obtaining an effective polish on nickel-free Cr-Mn-N, 18:8 Cr-Ni and 17 per cent Cr ferritic stainless steels; the current and voltage requirements, however, vary with the type of steel used.

## 2.0 Study of the Properties of Indigenous Foundry Moulding Materials

This project has been taken up with a view to assess the moulding characteristics of Indian foundry sands and bonding clays and to determine their suitability for various types of castings. A large number of samples had already been studied, and during the period under review ten sand samples were investigated, a brief account of which is given below:

(i) *Silica Sand* — This sand supplied by Messrs S. P. Foundries, Kanpur, was a fine-grained silica sand having an A.F.S. fineness No. of 108.8 and A.F.S. clay percentage of 4.9 per cent. The sand contained 79.80 per cent silica, 8.66 per cent  $Al_2O_3$ , 2.90 per cent  $Fe_2O_3$ , 0.48 per cent  $TiO_2$ , 1.56 per cent  $MgO$ , 1.40 per cent  $CaO$ , 3.6 per cent  $Na_2O$  and  $K_2O$ . The moulding characteristics of the sand were studied at different moisture contents ranging from 2.2 to 7.8 per cent after blending it with 5 per cent Bihar bentonite. A maximum green compressive strength of 6.95 lb./sq. in. at 2.6 per cent moisture content and a maximum permeability of 37 at 2.6 per cent moisture content were obtained. Optimum combination in respect of permeability and strength was obtained in the moisture range of 2.2-4.8 per cent. The sintering range of this sand was found to be 1150°-1200°C. Casting characteristics studied with respect to cast iron revealed poor surface finish with sandy layers adhering to the castings.



per cent ) in the presence of organic chemicals, involved in the manufacture of DDT, and of small amounts of chloride were also carried out. Preliminary results showed that the rapid failure of the heat-exchanger tubes is primarily due to the presence of water and HCl in the system.

#### **14.0 Production of Magnesium by Electrolytic Method from Magnesium Chloride**

Messrs Tata Chemicals, Mithapur, produce a large quantity of magnesium chloride as a byproduct in their salt industries which has a very limited market. Investigations on the electrolytic production of magnesium metal from this byproduct were pursued with limited success.

Subsequent to successful working of small electrolytic cells for electrolysis of fused magnesium chloride, a new design of cell for large-scale continuous work was taken in hand. Electrolytic experimental trials were carried out with separately designed cells to develop a continuously workable cell. It was noticed in one of the experiments that during the process of melting in the cell, the feed salt liberated considerable amount of gases setting the entire fluid in the cell in a turbulent motion, which subsided only when the volatile matters were completely driven out.

Three cells were designed with side chambers for localizing the turbulence, so that the electrolytic chamber could work smoothly and undisturbed. The results of the experiments, however, showed that unless the cell size was increased considerably, provision of such side chamber was not effective.

Experiments were carried out to find out what effect, if any, the amount of water, alkali chlorides and magnesium oxide in the cell feed would have on the anode and cathode surfaces during the electrolysis. From the results obtained it was found that if the feed chamber was not completely isolated from the electrolytic chamber, the presence of oxide built up an incrustation on the cathode

surface which prevented liberation of magnesium and agglomeration of any liberated magnesium. The liberated magnesium got dispersed throughout the molten mass, even in the vicinity of the anode. It was also found that the presence of water and alkali chlorides in the feed caused thickening of the electrolyte soon after starting up the experiments which practically prevented continuous work. Analyses showed presence of large quantity of magnesium oxide in the sludge.

The above difficulties can be solved by the following steps:

1. Use of a large-size electrolytic cell or use of a separate premelting pot for magnesium chloride.
2. Modification of the drying procedure by drying the cell feed without the use of ammonium chloride.

In order to give shape to the above idea a cell has been designed with completely isolated melting and electrolytic chambers. The cell is now in fabrication stage.

Further, the drying procedure has been changed to avoid excess of ammonium chloride in finished feed salt. Since the newly designed cell will require nearly 300-400 lb. of salt for a single trial, a larger drying oven has been designed, which is also in fabrication stage.

#### **15.0 Liquid Gold**

Liquid gold is widely used by the glass and pottery industries for decoration purposes. Almost the entire demand of this material is met from imports. The project has been taken up with a view to prepare liquid gold of high quality from indigenous materials.

A process has been developed for producing good quality liquid gold and some samples were sent for trial and report. The method has been handed over for commercial exploitation to several firms on payment of handsome premia and production royalties.

## 16.0 Preparation of Plating Salt

Almost all plating salts are imported in India today. The investigation was taken up with a view to prepare different plating salts from indigenous material.

(i) *Nickel - plating Salts* — Experiments were carried out on the formulation of dull nickel-plating solution. A nickel-plating bath of the following composition was prepared in the laboratory:

Nickel sulphate	200 gm./litre
Nickel chloride	45 gm./litre
Boric acid	30 gm./litre
Operating conditions:	
pH	5.6-5.8
Temperature	105°-110°F.
Current density	20-40 amp./sq. ft.

Bath preparation was carried out in a rubber-lined tank. The bath did not work satisfactorily in the beginning and the deposit was pitted and off colour. These conditions were remedied by working the bath for some time. Electrolysis at low current density (2-3 amp./sq. ft.), using corrugated steel sheets as cathodes, removed the objectionable impurities generally associated with the main chemicals and put the bath in operating conditions.

The bath had good throwing power and high current efficiency and gave satisfactory nickel deposits.

(ii) *Copper-plating (Cyanide Bath) Salts* — Experiments were carried out on the formulation of copper-plating salt. The copper solutions prepared in the laboratory gave good copper deposits and a high cathode current efficiency. Bath preparation was carried out in a 5-litre beaker. The requisite amount of potassium cyanide was dissolved in water and was filtered and used for copper-plating in a 5-litre beaker. The anodes were pure electrolytic copper and were bagged. Steel or brass panels, each 3 × 1 in., were used as test panels. The various formulations varied in their cuprous cyanide content from 15 gm. to 25 gm./litre and potassium cyanide varied between 30 and 40 gm./litre.

The copper deposits showed excellent adhesion on mild steel and brass test-pieces. There was no roughness and blisters were not encountered. The increase in temperature resulted in an increase in the current efficiency. Increase in current density resulted in a decrease in the cathode current efficiency. The bath gave higher current efficiency in comparison with the "Cannings Zonax copper salt". Copper cyanide baths were also prepared using sodium cyanide in place of potassium cyanide and the results obtained were extremely satisfactory.

Experiments were carried out with a view to prepare cuprous cyanide from commercial copper sulphate and sodium cyanide. Pure cuprous cyanide thus prepared has been successfully used in copper cyanide baths.

## 16.1 Deposition of Copper on Mild Steel Wire

Copper deposition by immersion may be used for decoration or as a lubricant, e.g. in drawing steel wire or forming sheet metal. The process is also useful for tyre-beading wire. In the absence of technical data for this purpose, work was undertaken to develop a suitable bath to deposit copper by immersion on steel wire. The copper coating should be bright and adherent after drawing. The process consists in producing a copper coating by immersing the cleaned steel wire in acid copper sulphate solution. A large number of experiments were carried out on mild steel coils by immersion in acid copper sulphate bath containing stannous chloride and glue. The coils of wire were properly cleaned, pickled in 10 per cent sulphuric acid and given a bright dip in chromic acid solution before immersion in the copper solution. The composition of the bath was:

Copper sulphate	25 gm.
Sulphuric acid	60 ml.
Stannous chloride	5 gm.
Sodium chloride	20 gm.
Glue	5 gm.

Water to make 1 litre.

Immersion time varied from 2 to 5 minutes. The deposit flaked off, if the immersion time exceeded 5 minutes. The process requires careful manipulation. Maximum iron content tolerated in the bath is 5 per cent after which it is rejected. Satisfactory copper deposits on mild steel coils have been obtained and even after drawing the coatings are firmly adherent. The final colour of the coating depends on the technique of "drawing". Bright finish can be obtained by "wet drawing" the above coated wire. For tyre manufacture, "dry drawn" copper-coated steel wire is suitable.

### 17.0 Determination of Gases in Metals

The presence of gases in metals is known to influence their properties and at times rather adversely. Gas analysis of metals, therefore, forms an important part in research on metals and alloys. The apparatus required for this purpose is very costly and requires to be imported. Work has, therefore, been taken up in fabricating apparatus for accurate determination of gases and to undertake a systematic analysis of oxygen, nitrogen, hydrogen, etc., in metals and alloys.

The vacuum fusion apparatus, which had been designed and fabricated with all its accessory parts in the National Metallurgical Laboratory, is working most efficiently for the determination of oxygen, nitrogen and hydrogen in metals and alloys with great accuracy. The heating is performed in a 6 kW. spark-gap high-frequency furnace working at a frequency of 30 kilocycles per second. Melting of the sample is carried out in a graphite crucible at a temperature of 1550°-1600°C. The degassing of the crucible is carried out at a temperature of 1900°C. for a period of 3 hours. Maintenance of high vacuum ( $10^{-5}$  mm.) and low blank (0.25 c.c. per hour) are the salient features of the apparatus. Various metals and alloys were accurately analysed for oxygen, nitrogen and hydrogen during the period.

Various samples such as electrolytic manganese, coinage alloys, rail steel, boiler plate, electrolytic ferro-chrome, etc., were also analysed for hydrogen in the vacuum hydrogen determination apparatus which was earlier fabricated in the National Metallurgical Laboratory. Both vacuum fusion apparatus and the vacuum heating hydrogen determination apparatus have saved foreign exchange worth Rs. 60,000.

### 18.0 Spectrographic Studies

(i) *Development of a Suitable Spectrochemical Method of Determination of very Low Percentage of Boron in Steel* — Industrial use of boron as an alloying element in steel is rapidly increasing and its presence in very low percentage makes it difficult for accurate determination by chemical method. Spectrochemical method has, therefore, been tried and standardized. To obtain the required high sensitivity and accuracy in the determination of boron, rotating electrode serving as lower electrode was employed. It was allowed to rotate in the solution of the sample kept in a small porcelain boat so as to ensure a continuous feeding of the solution on the electrode during excitation. A.C. intermittent arc after K. Pfeilsticker circuit was used for excitation. The intensity ratio of the boron line (2496.78Å) and the iron line (2947.3Å) were determined photometrically. The concentration of boron was read from the analytical curve relating log deflection ratio to log concentration. Synthetic standards were prepared by solution method to make the standard working curve. Estimation up to 0.001 per cent of boron is possible by the method and the results obtained are quite satisfactory.

(ii) *Spectrographic Estimation of High Percentages of Chromium and Manganese in Nickel-free Austenitic Stainless Steel* — Work has been taken up to develop a suitable and rapid method of simultaneous analysis of high chromium and manganese in



nickel-free austenitic stainless steel spectrographically.

Spectrographic determination of high percentage of elements present in the sample often offers difficulty in getting accurate results. Nickel-free stainless steel developed at the National Metallurgical Laboratory contains high Cr and Mn. Conventional chemical methods, though accurate, are time-consuming. Efforts have been made to develop a method so that high Cr and Mn contents of nickel-free stainless steel can be determined spectrographically very quickly whilst attaining same degree of accuracy like the chemical method. Experiments were made to reduce the intensity of Cr and Mn lines by using neutral filters of various densities with large quartz spectrograph and finally filter of density 0.99 proved to be quite satisfactory to give the required analytical line pairs of Cr and Mn suitable for microphotometer measurements with a considerable reduction of background also. Porous cup sparking technique, with samples in solution form, was adopted. Synthetic standards of variable composition (Cr, 15-25; Mn, 3-15; and Cu, 0.5-3.0 per cent) were prepared and a few exposures were taken under standardized conditions to study the intensity of the lines to be used for analytical purpose. The working curves for both Mn and Cr were plotted with the data obtained from the above conditions, and it was observed that Mn curve was quite smooth and linear without any variation whatsoever, but working curve for Cr above 20 per cent appeared to be fluctuating. Further investigations to rectify these irregularities are in progress. This method also gives scope for the determination of Cu that may be present in these types of steels.

(iii) *Spectrographic Estimation of Phosphorus in Steel* — Investigation has been taken up to develop a method to estimate phosphorus in steel spectrographically using Hilger large quartz spectrograph.

Attempts were made to make use of the existing Hilger large quartz spectrograph in

estimating P in steel. In the case of large quartz spectrograph, P 2136.2A line had to be selected for analysis. Cu 2135.98A is an interfering line with P 2136.2A line. This difficulty will not arise at all provided the steel is absolutely free from copper. Experiments were conducted with a series of exposures of standard samples of various phosphorus and copper contents (up to 0.46 per cent Cu). In case of 0.46 per cent Cu only, line of copper 2135.98A came along with P 2136.2A but was well separated from P 2136.2A line. Spectrographic plates of high sensitivity were used, and to get better excitation rotating electrode was employed with very satisfactory results. Intermittent A.C. arc of 15 kW. was used for excitation. Phosphorus line up to 0.02 per cent phosphorus content was well obtained. Further progress could not be made for want of correct type of spectrographic plates.

(iv) *Rapid Spectrographic Method for Analysis of Slag* — Investigation has been taken up to develop a suitable routine spectrographic process for the analysis of steel-making and blast furnace slag.

To start with, basic open-hearth slag was taken and the sample was fused with a mixture of sodium carbonate and borax (all spectrographically pure) at about 1000°C. Finally, this mass was extracted with 25 per cent HNO<sub>3</sub> and a few c.c. of hydrogen peroxide solution. A very clear solution of the sample was obtained. Large quartz spectrograph was employed. A few c.c. of the solution were dried on graphite electrode (lower electrode). A.C. intermittent arc was used for excitation, taking graphite as counter-electrode. For estimation of CaO and SiO<sub>2</sub>, neutral filter of density 0.99 (i.e. 10 per cent transmission of the radiation) was used. The prominent analytical lines of Si and Ca were obtained with a satisfactory reduction of intensity of the lines suitable for microphotometer measurement. For the determination of Al<sub>2</sub>O<sub>3</sub>, MnO and MgO, however, filter need not be used.



## 19.0 Beneficiation of Low-grade Manganese Ore

(i) *From Lohandabud Group of Mines, Jharsuguda, Orissa* — The investigation was sponsored by Messrs Shivji Nathubhai of Jharsuguda to beneficiate their low-grade manganese ores so as to make them suitable for ferro-manganese production.

Three samples of low-grade manganese ore (marked La, Lc and Ld) from Lohandabud group of mines, Jharsuguda, Orissa, were mixed together for purposes of beneficiation, as these were similar in nature, i.e. low in iron and high in silica. The mixed sample assayed: Mn, 40.02;  $\text{MnO}_2$ , 34.71; Fe, 5.66;  $\text{SiO}_2$ , 19.34;  $\text{Al}_2\text{O}_3$ , 3.8; CaO, 1.1; BaO, 3.32; MgO, 0.69 and P, 0.18 per cent. Petrological examination of the sample showed that braunite was the principal manganese mineral followed by psilomelane, hausmanite, pyrolusite, jacobsonite, hollandite, sitapelite, and vredenburghite, while quartz, garnet and pyroxenes constituted the chief gangue followed by amphiboles, barytes, biotite, feldspar and haematite. Fair liberation of the gangue occurred at about 100 mesh.

High intensity magnetic separation of the mixed sample after grinding to —35 mesh and desliming yielded a manganese concentrate assaying Mn, 43.3; Fe, 5.8; and  $\text{SiO}_2$ , 13.2 per cent, with a recovery of 71.7 per cent Mn. Electrostatic separation of the magnetic product obtained from high intensity magnetic separation after hydroclassification of the —35 mesh ground sample gave a concentrate assaying Mn, 45.2; and  $\text{SiO}_2$ , 9.7 per cent, with a recovery of 63.4 per cent Mn. Cationic flotation at a pH range of 6.5-7.0 yielded a manganese concentrate assaying Mn, 45.1 and  $\text{SiO}_2$ , 10.5 per cent, with a recovery of 51.2 per cent Mn.

As the composite sample did not yield satisfactory results, a few experiments were carried out with the individual samples to study whether a suitable concentrate could be obtained. Satisfactory results could not be achieved with two samples, namely La

and Lc, because of their very complex nature. Sample Ld, when subjected to high intensity magnetic separation, yielded a magnetic product (manganese concentrate) assaying Mn, 47.3; Fe, 6.0;  $\text{SiO}_2$ , 10.7;  $\text{Al}_2\text{O}_3$ , 3.4; S, 0.06; and P, 0.18 per cent.

(ii) *Briquetting and Sintering Studies of Manganese Concentrate from Low-grade Ore from Siljora-Kalimati Mines, Orissa* — The project was taken up on behalf of Messrs Rungta & Sons, Chaibasa, for preparing manganese concentrates from the low-grade manganese deposits of their mines and for conducting studies on their briquetting and sintering characteristics so as to make them suitable for charging directly in the furnace for the production of ferro-manganese.

About 5 tons of low-grade manganese ore were crushed to  $-1\frac{1}{2}$  in. size and subjected to reduction roast treatment in a vertical reduction furnace for preparing manganese concentrate for briquetting and sintering studies. Dry magnetic separation, at —4 mesh, of about  $3\frac{1}{2}$  tons of the reduced ore was carried out. Wet magnetic separation of the magnetic product obtained from the above test, after grinding to about 65 mesh, yielded a combined manganese concentrate assaying Mn, 49.5 and Fe, 7.9 per cent, with a manganese recovery of 69.4 per cent.

Laboratory studies were made on the sintering characteristics of the manganese concentrate. The effects of variables such as coke, moisture and sinter fines in sinter mixture on the sintering time and on the quality of sinter produced were studied. Research results showed that the optimum amounts of coke and water contents were 6.0 and 10.0 per cent respectively for producing a sinter with good size stability. The final sinter assayed Mn, 53.46; Fe, 8.13;  $\text{SiO}_2$ , 3.42;  $\text{Al}_2\text{O}_3$ , 10.19; and P, 0.055 per cent.

## 20.0 Beneficiation of Gypsum from Rajasthan

A sample of low-grade gypsum assaying 37.71 per cent  $\text{SO}_3$  (gypsum equivalent to

81.08 per cent) was received from Messrs Bikaner Gypsums Ltd., Rajasthan, for beneficiation purposes. The sample contained quartz and calcite as chief gangue minerals followed by minor amounts of mica, hornblende, chlorite, tourmaline, garnet, feldspar, halite and clay minerals.

Flotation tests under varying conditions were performed with the sample with a view to produce a concentrate of plaster of paris grade. Use of sodium oleate as collector for gypsum, sodium silicate as a depressant for siliceous gangue and pine oil as a frother at a pH of 10.0 produced a concentrate assaying 95.5 per cent gypsum, with a recovery of 91.2 per cent. Further work is in progress.

## 21.0 Beneficiation of Fluorspar from Thurwali, Rajasthan

Fluorspar is an important raw material for the manufacture of steel, aluminium and other non-ferrous metals, chemical, glass and ceramic ware, etc. At present the country's entire requirement is met by import. This investigation has been taken up for the Director of Mines and Geology, Government of Rajasthan, to beneficiate the low-grade fluorspar deposits at Rajasthan so as to make them suitable for various industrial applications. The sample as received assayed:  $\text{CaF}_2$ , 22.5;  $\text{Fe}_2\text{O}_3$ , 2.7;  $\text{SiO}_2$ , 64.25;  $\text{Al}_2\text{O}_3$ , 4.3; Pb, 0.04; and S, 0.2 per cent. Flotation employing oleic acid as collector for fluorspar and sodium silicate as a gangue depressant produced a concentrate assaying 70.0 per cent  $\text{CaF}_2$ , with a recovery of 95.8 per cent. Use of methyl isobutyl carbinol as a frother yielded a rougher fluorspar float assaying 68.0 per cent  $\text{CaF}_2$ , with a recovery of 98.6 per cent. Flotation of the sample ground to different periods, namely 11, 13 and 15 minutes, in a laboratory rod mill, using sodium oleate and sodium silicate at a pH of 7.2, yielded rougher fluorspar floats assaying 71.9, 71.9 and 65.8 per cent  $\text{CaF}_2$ , with recoveries of

91.6, 92.3 and 93 per cent, respectively. Since 15 minutes' grind yielded the maximum recovery, this grind was employed for subsequent flotation tests. Flotation tests performed with sodium hydroxide and soda ash as pH regulators did not yield better results.

Use of distilled water (pH 6.8) in place of soft water with 1.5 lb. per ton of sodium silicate and 0.75 lb. per ton of sodium oleate for flotation yielded a rougher float assaying 76.9 per cent  $\text{CaF}_2$ , with a recovery of 92.7 per cent. The rougher float, when cleaned twice, yielded a refloat concentrate assaying 88.9 per cent  $\text{CaF}_2$  representing a recovery of about 88.0 per cent. Further work is in progress.

## 22.0 Reduction of Iron Content in a Bauxite Sample from Jabalpur, M.P.

Presence of iron oxide, etc., makes bauxite unsuitable for direct application in metallurgical, refractories and other industries. The investigation was taken up on behalf of Messrs Associated Cement Companies for the reduction of iron and titanium oxide in their low-grade bauxite sample from their mines in Jabalpur district, M.P.

The sample as received assayed.  $\text{Al}_2\text{O}_3$ , 53.3;  $\text{SiO}_2$ , 6.31;  $\text{Fe}_2\text{O}_3$ , 5.20;  $\text{TiO}_2$ , 8.0;  $\text{CaO}$ , 0.25 and loss on ignition, at  $950^\circ\text{C}$ ., 26.83 per cent. High intensity magnetic separation of the sample at -10 mesh after screening into two fractions yielded a non-magnetic product assaying 3 per cent  $\text{Fe}_2\text{O}_3$  and 7.2 per cent  $\text{TiO}_2$ . Magnetic separation at -48 mesh did not improve the results. Tabling the sample at -20 mesh after classification produced a bauxite concentrate assaying 3.5 per cent  $\text{Fe}_2\text{O}_3$  and 7.5 per cent  $\text{TiO}_2$ . The -3 mesh original sample was subjected to reduction roast treatment at  $520^\circ$  and  $550^\circ\text{C}$ . for 15 and 30 minutes respectively. Magnetic separation of the reduced sample was completed. Further work is in progress.

## 23.0 Production of Basic Refractories from Indigenous Sources

(i) *Studies on Almorah Magnesite* — Magnesite bricks are indispensable in lining the basic open-hearth furnaces. The most well-known magnesite deposit in India occurs at Salem in Madras. Recently new deposits have been located in Almorah district of U.P. Laboratory scale study conducted previously on these samples indicated that this magnesite, similar to the Austrian type, could be more advantageously used for the production of magnesite bricks than that of the deposits at Salem.

Large-scale batch trials on a semi-pilot plant scale were undertaken on two tons of representative sample from Agargiri-chchina in Almorah district. The sample ores differ in composition from that of Doba area and contain a small amount of dolomite. Initial calcination studies showed that —5 mesh samples calcined to 1500°C. contained free lime which hydrated on exposure. Even calcination at 1600°C. showed the presence of a small amount of free lime and this was attributed to inhomogeneity in the distribution of lime. Studies on the grain size versus temperature of firing showed that lime was completely stabilized at 1500°C. on samples made from —36 mesh size or finer. With higher firing temperature stabilization of lime could be done at a coarser mesh size. The deductions were arrived at after a careful study of the hydration expansion, mineral constitution and specific gravities of the calcines. Further work on the production of a ton of magnesite bricks and testing their properties is being taken up.

(ii) *Tar-bonded Dolomite* — Dolomite is one of the important refractory material, used in steel-making furnaces like Bessemer converter, L.D. converter and basic open-hearth. Some of the newly built steel plants in the public sector envisage the use of tar-bonded dolomite in Bessemer and L.D. converters. With a view to study the suitability of Indian dolomite for semi-stabilization for the

above purpose, samples were collected from various parts of the country including Bhilai and Rourkela. Hydration-resistance of the fired dolomite of different grain sizes was determined with additions of mineralizers such as iron oxide, clay and titania. The best mineralizers for optimum sintering and hydration-resistance were determined. Experiments were conducted on the suitability of different types of tars and pitches for bonding the sintered dolomite. Tar-bonded dolomite as per the above findings was rammed in a small cylindrical vessel. The lining was hardened by firing it with coke and oxygen. After pouring the hot metal into the vessel, oxygen was blown through the metal by lancing. The lining stood the test well, and even after cooling, it did not hydrate for two weeks. The lining is expected to give good life on continuous runs.

(iii) *Chrome - magnesite Refractories* — In view of the expansion of the iron and steel industry during the Second and Third Plan periods, the demand for basic refractories using chrome ore is expected to steadily increase. Recent trends in steel-making and refractory practice in all basic open-hearth furnaces having chrome-magnesite roofs have clearly indicated their superiority in respect of life of the lining, operational temperature and increased steel production.

With a view to assess the usefulness of Indian chrome ores for refractory purposes, samples were obtained from different localities and chemical analysis and mineralogical studies were carried out. The chromic oxide content of the ore ranged from 38 to 48 per cent, iron oxide 18 to 28 per cent and silica 6 to 9 per cent. These were composed of chromite and gangue minerals such as serpentine, talc, pyroxene, plagioclase feldspar and secondary iron oxide. After a preliminary study, three samples of chromite from Mysore, Bezwada and Bombay State were selected for detailed investigation. Calcined magnesite was added in different grain sizes and the mix having maximum packing density was



selected. The relationship between the grain size of chrome ore and its iron oxide bursting tendency and spalling-resistance was studied. Grain size being equal, the iron oxide content of the ore appeared to have significant influence on the bursting tendency. Physical properties such as refractoriness under load, porosity, spalling-resistance and linear expansion, etc., were determined. Further work to develop a suitable composition possessing optimum properties is in progress.

#### **24.0 Development of Refractories from Travancore Beach Sand**

(i) *Zircon Refractories* — With the rapid growth of metallurgical industries such as the aluminium industry during the Second Five Year Plan as also the glass industry, it is expected that there will be a considerable demand for zircon refractories which are widely used in aluminium remelting furnace, glass tank furnace, etc. This product is not manufactured at present in India. In view of the abundance of raw material available from Travancore beach sand, this project was initiated for the development of zircon refractories utilizing zircon which is obtained as a byproduct in the processing of beach sands of Travancore-Cochin. Semi-pilot plant studies on the manufacture of zircon bricks and special shapes from the compositions finalized in the earlier investigation were completed. A comprehensive report on the development of these refractories was prepared. Studies on the load-bearing property of various phosphate-bonded refractory compositions indicated that bodies cured at low temperatures were found to fail around a temperature of  $1450^{\circ}\text{C}$ . (ta), although they develop a good strength and bulk density on curing between  $250^{\circ}$  and  $400^{\circ}\text{C}$ .

(ii) *Sillimanite Refractories* — Sillimanite is also obtained as a byproduct in the processing of the beach sand of Travancore-Cochin. The project has been initiated to utilize sillimanite for the production of sillimanite refractories which will reduce the cost of

production as compared to the present-day method of manufacturing this material from kyanite.

Studies were made on the modification in the body compositions and their mode of preparation which have shown that these refractories can be manufactured from the Travancore beach sillimanite sand by (i) ageing the clay slip before mixing it with sillimanite sand and ball mill fines, and (ii) by employing  $\text{MgO}$  (3 per cent) as mineralizer.

A combined scheme for the manufacture of zircon as well as sillimanite refractories from the beach sands of Travancore has been drawn up.

#### **25.0 Development of Superduty Silica Bricks**

With the expansion of metallurgical and related industries during the successive Five Year Plans the demand for superduty silica bricks from indigenous raw materials has increased. A number of quartzite samples collected from different localities were studied and Jamda quartzite was selected for further investigation. A consignment of 5 tons of quartzite from Jamda was received for carrying out large-scale trials. As a result of packing density studies of graded Jamda quartzite mixes, the porosity of silica brick specimens was brought down to below 20 per cent and one lot of full-size bricks fired in the down-draft kiln produced bricks of good crushing strength, porosity below 20 per cent and good refractoriness under load. In another lot fired in a similar way, the refractory bricks did not develop sufficient strength. The cause of this defect is being studied and experiments on small-size buttons are being planned to standardize a proper firing schedule so that the specimens develop good bond and possess adequate strength.

To study the effect of various mineralizers like  $\text{CaO}$ ,  $\text{BaO}$ ,  $\text{MgO} + \text{MgCl}_2$  on the conversion of Jamda quartzite, the above mineralizers were added in different proportions to graded Jamda quartzite with and without



the addition of iron oxide and a large number of 2 in. diameter specimens were pressed at different pressures. One batch of specimens was fired on a predetermined slow schedule and most of the specimens came out of the kiln without any cracks. The physical properties of these specimens are under study.

### 25.1 Studies on Dhanji Hill Quartzite

This quartzite was sent by the Geological and Mining Department of the Government of Bihar to study its suitability for silica refractories. Its alumina content was rather high, being 2.22 per cent, and the sample as received was mixed with basic rocks. Full-size bricks were made with this material using 45-10-45 grading with 2 per cent lime as bond and 1 per cent mill scale as mineralizer. The bricks were fired along with other silica brick specimens in the down-draft kiln on a predetermined schedule. It was observed that the bricks were completely vitrified, fused and deformed. Though this led to the preliminary conclusion that the material will not be suitable for silica brick, further small-scale trials were carried out after a careful selection of the good quality quartzite and exclusion of all basic rocks mixed therein. Specific gravity and porosity of the raw rocks as well as after firing at 1450°C. were determined and silica brick specimens were prepared with 2 per cent lime and with or without 1 per cent mill scale as mineralizer. This study revealed that though this material

would not be suitable for making open-hearth roof quality silica bricks, by careful removal of basic rocks and other impurities and properly controlled processing, it may be possible to utilize this material for making silica bricks for use under less severe conditions.

### 26.0 Study on Soderberg Paste

Considerable tonnages of electrode paste are imported for the manufacture of electrodes used in electro-metallurgical industries. With the establishment and expansion of India's ferro-alloy and aluminium industries, demand for the electrode paste will increase manifold. This project has been initiated with a view to produce carbon paste of superior quality from dense carbon aggregates made out of indigenous raw materials. During the period under review, indigenous pitches and tars were studied for developing a good binder. Studies were also made in determining packing characteristics of dense carbon aggregate powders. Based on the data thus obtained, different aggregate binder mixes were compounded and their physical properties, both in the raw as well as in the baked state, were determined. Some of these mixes proved to be quite suitable for Soderberg paste. Full details of these compositions and the methods of producing the same are the content of a patent application. The properties of a few compositions and the properties of a well-known imported paste are given in the following table:

**TABLE 1—PHYSICAL PROPERTIES OF THE EXPERIMENTAL AND FOREIGN (FRENCH) TAMPED CARBON PASTES**

SL. No.	DETAILS OF CARBON PASTE	GREEN PROPERTIES: MEAN MODULUS OF RUPTURE kg./cm. <sup>2</sup>	BAKED (1200°C.) PROPERTIES				ELECTRICAL RESISTIVITY AT ROOM TEMP. ohms-centimeter
			Mean Compressive Strength lb./sq. in.	Mean Modulus of Rupture kg./cm. <sup>2</sup>	Vol. Shrinkage	Porosity %	
1.	*Experimental paste	6.32	2017	59.2	Less than 1%	22-23	1.5 { $0.57 \times 10^{-2}$ to $0.65 \times 10^{-2}$
2.	do	21.46	1470	49.0	do	23-24	n.d. $0.11 \times 10^{-1}$
3.	French paste	46.90	1411	53.3	Not determined	24-26	n.d. $0.18 \times 10^{-1}$

\*Different binders were used.

## 27.0 Clay-bonded Graphite Crucibles

The work on this project was patented and has already been handed over to industries for commercial production on payment of handsome premia and production royalties. The problem was to develop a suitable body composition from indigenous materials possessing adequate strength as well as thermoplasticity at the metal founding temperatures with a glaze that should form a tenacious and protective coating over the crucible surface. Both these objectives were successfully achieved.

## 28.0 Development of Calcium-aluminate Cement

The project has been taken up with a view to develop calcium-aluminate cements using indigenous raw materials for the manufacture of castable refractories.

(i) *Mono-calcium Aluminate* — A reverberatory furnace with a screw feeder was designed and fabricated at the laboratory and trial runs were made with uncalcined batches consisting of ferruginous bauxite and limestone. This furnace has a melting chamber of about 18 in. square with a pre-calcining zone of 21 in. It is fitted with an automatic feeder which can maintain a feed of  $1\frac{1}{2}$  lb. of material per minute. This feed can be varied. The furnace reached  $1450^{\circ}\text{C}$ . in about 5 hours and then a charge of 20 lb. was fed into the furnace. Once the temperature was reached and maintained, a continuous flow of melt at the rate of  $\frac{3}{4}$  lb. per minute was obtained. It is hoped that this furnace with little modifications can be used for the continuous production of calcium-aluminate cements from bauxite and limestone.

(ii) *Calcium-dialuminate* — Forty calcium-dialuminate cement compositions were made using either pure chemicals or raw materials on the basis of molar weights and were sintered at  $1650^{\circ}\text{C}$ . (3 hours), crushed and powdered to -100 mesh. Specific gravity determinations of these compositions were

completed. Setting behaviour and percentage of gauging solutions required to make them workable were studied. A comprehensive microscopic study was taken up. Samples were prepared by sintering twice in order to complete the solid state reactions and these are under study.

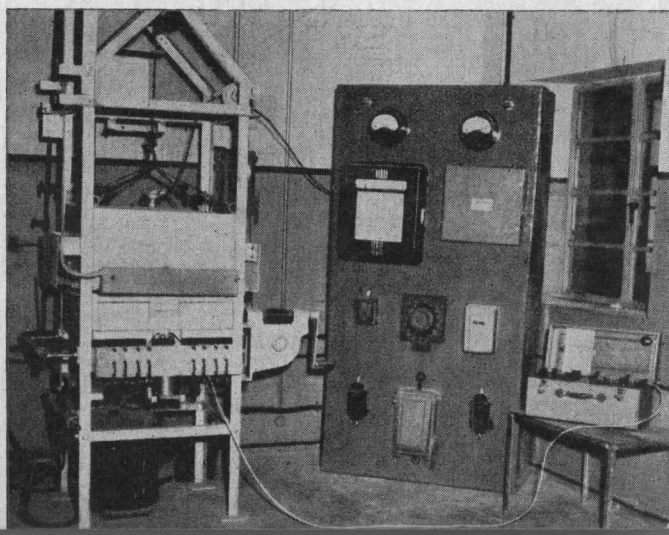
## 29.0 Studies on Clays from Bansi, Mirzapur District

The study of the suitability of the two samples of clays from Bansi for refractory purposes was completed and a report on the work done along with suggestions for commercial use of the clay was sent to the Director of Geology and Mining, U.P. Both the clays were found highly refractory and required a firing temperature of not less than  $1450^{\circ}\text{C}$ . to produce refractories with suitable properties. Proper blending with more fusible clay has been suggested for the utilization of these clays for medium-heat duty class of refractories. On account of the fairly high refractoriness under load of the white clay, the possibility of its use for high-heat duty refractories has also been suggested by suitable blending and processing techniques.

## 30.0 Thermal Conductivity of Refractory Materials

The thermal conductivity apparatus designed and fabricated at the National

THEMAL CONDUCTIVITY APPARATUS FABRICATED AT  
THE NATIONAL METALLURGICAL LABORATORY FOR  
THERMAL CONDUCTIVITY DETERMINATION OF RE-  
FRACTORY MATERIALS



Metallurgical Laboratory as per A.S.T.M. specifications was standardized and regular work on collection of thermal conductivity data on different types of insulation refractories was started. The data collected will be used in collaboration with the Indian Standards Institution for formulating specifications for insulation refractories.

### **31.0 X-ray Studies on Refractory Materials**

Preliminary investigation was undertaken on the suitability of the available equipment for the determination of lattice of crystals of ceramic materials with a cubic structure by the Debye-Scherrer method. A Unicam. 6 cm. camera with a goniometer head was used and Straumanis or asymmetric mounting of the film was adopted as this avoids corrections for film shrinkage.

Debye-Scherrer powder pattern of three chromite ores was taken using both  $\text{CoK}\alpha$  and  $\text{CuK}\alpha$  radiation. In some cases, fluorescence due to absorption was considerable. The use of an aluminium foil between the film and the diffracted rays lessened the background effect due to fluorescence. Pure chrome grains were isolated by elutriation and X-ray pictures were taken. Solid solution effects in chromite by different additions and firing treatment are under study.

### **32.0 Base Exchange Studies on Indian Clays**

Cation exchange on clays offers a diagnostic method of identifying clay minerals present in a particular clay, as the type of ions absorbed on the clay affects the green and fired properties of clays. Much work has been done on the effect of adsorbed cations such as  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Li}^+$ ,  $\text{Ca}^{++}$ ,  $\text{Ba}^{++}$ ,  $\text{Mg}^{++}$ , etc., on the workability of clays. It is contemplated in the present work to carry out some specific cation exchange on some Indian ceramic clays and study its

influence on the green and fired properties of clays. It is also contemplated to carry out differential thermal analysis and X-ray analysis as supplementary to base exchange in identifying the clay minerals present. With this object in view, about ten clays, consisting of fireclays, bond clays, china clays and bentonites, were obtained and six of them were crushed to pass 18 mesh B.S.S. and are being analysed after sampling by standardization. The sieve and subsieve analyses of two clays were completed and the analyses of the rest of the sample are under progress. Size analysis is undertaken in order to know the relative fineness of the various clays.

Since clay minerals are of the dimensions of about 1 micron and below, it is necessary to isolate a fraction finer than 2 microns in order to study the cation exchange capacity characteristic of a particular clay mineral. Hence isolation of a fraction finer than 2 microns of Kotah fireclay was undertaken as a prerequisite for determining the base exchange capacity. The particle size determination apparatus of Gallenkamp was put into working condition and the size analysis of the clays with this apparatus was found to be unsatisfactory for these materials and hence it was abandoned. A column technique for determining the base exchange capacity of clays with ion-exchange resin Amberlite IR-120(H) is under contemplation and a suitable column is being devised.

### **33.0 Development of Magnetic Materials**

The demand for magnetic materials is multiplying in India following establishment of telephone, electronics and other industries. At present the quantity needed is mostly met by import. This project has been taken up with a view to obtain detailed technical knowledge regarding their manufacture and to develop, if possible, permanent magnets and other magnetic materials from indigenous



sources. The work is initially concerned with the preparation and study of alloys of the well-known Alni, Alnico and Alcomax types.

An appreciable quantity of permanent magnets was prepared as a preliminary for the semi-commercial production of cast permanent magnets of the Alni, Alnico and Alcomax types on which extensive work had hitherto been done. A detailed scheme was prepared for such semi-commercial production. The development of permanent magnets of these types, starting from powders of the constituent metals and using the techniques of powder metallurgy, has also been taken up.

One or two specific compositions in the range of the low-aluminium, low-manganese alloys previously studied gave encouraging low values of hysteresis loss and coercive force, indicative of its applicability in transformers and other electrical machinery. Further work on these alloys was carried out and a set-up for both vacuum as well as hydrogen annealing at fairly high temperatures was assembled. The commercially available hydrogen has a rather high proportion of oxygen and considerable efforts were made with fair success to practically eliminate the residual oxygen from the commercial hydrogen.

The magnetic testing of pressed and sintered powder compacts presents certain new problems for which construction of appropriate equipment becomes necessary. Magnetic testing of both the cast Alni and Alnico alloys as well as the low-manganese, low-aluminium transformer grade steels had been hitherto carried out on cylindrical test specimens, with length to diameter ratio exceeding 25, so that the demagnetizing factor is both small and can be accurately calculated. It will obviously not be possible to prepare similar specimens from pressed and sintered powder compacts and it will be necessary to use test specimens which may be both not cylindrical and having a  $L/D$  ratio smaller than 5, with correspondingly large demagne-

tizing factors. Test experiments were, therefore, performed using very short samples with the present circuit and experimentally determining the accuracy of such measurement. The design and construction of a new set-up, specifically for short magnets, were also taken up. For the latter, a powerful electromagnet, which was recently received, was used and preliminary studies using this set-up are being carried out.

#### **34.0 Preferred Orientation in Rolled Sheets**

The usual method of determining the preferred orientation produced on cold-rolled sheet is based on the study of the nature and intensity of X-ray reflections. This method could advantageously be replaced, if possible, by methods based on the values of the electrical conductivity and the velocity of propagation of stress waves in rolled sheet as a function of the angle to the direction of rolling.

A ballistic method for the determination of the anisotropy of electrical resistivity of cold-rolled sheet was devised and standardized and it has been established that an accuracy of about 0.2 per cent can be obtained without considerable difficulty. Concurrently with the determination of electrical resistivity by this method, electrical resistivities of both cold-rolled aluminium and cold-rolled brass as a function of the angle between the direction of measurement and the direction of rolling were determined using the standard Kelvin Double Bridge method. A small but unmistakable anisotropy in the electrical resistivity was detected in both cases, in line with recent experimental work in this field, where such unexpected anisotropy values are being related to the density of dislocations in the various crystallographic planes and directions in a structure having preferred orientation. The values of the electrical resistivity obtained with brass are given in Table 2. It is seen that they are in most cases mutually consistent and

**TABLE 2 — ELECTRICAL RESISTIVITY  
OF COLD-ROLLED BRASS**

ANGLE BETWEEN SPECIMEN LENGTH AND DIRECTION OF ROLLING	SPECIFIC RESISTANCE (IN $10^{-6}$ OHM/CM.)	
	Cold Reduced 95%	Cold Reduced 98%
0	7.53	8.57
90	7.17	8.04
12	7.64	9.03
22½	7.20	8.11
34	7.59	8.03
45	7.51	8.49
57	7.61	8.14
67½	7.00	7.69
79	7.52	7.76

Specific resistance for  
annealed material:

$6.84 \times 10^{-6}$  ohm/cm.

a further examination of the anisotropy is warranted.

### 35.0 Mechanical Properties of Stressed Materials

The object of this investigation is to study by resonance methods the velocity of propagation of acoustic waves of medium to high frequency on previously stressed test-pieces and to examine if changes in the mechanical properties that arise can be determined therein.

The elastic constants of a wide variety of commercial metals and alloys were determined. When the required metals or alloys were not available in a suitable form for study by this method, small ingots were made and suitable specimens prepared by necessary forging and grinding. Data had been collected on many of the common metals and on a range of plain carbon steels. Data were also obtained on metals like bismuth and tin, on which they are generally meagre and not comprehensive.

### 36.0 Structure of Carbides in Alloy Steels

The physical and mechanical properties of alloy steels greatly depend upon the nature

and amount of carbides present in them. These, in turn, are influenced by the nature and amount of alloying elements present. This investigation has been taken up with a view to study the nature and structure of carbides on a wide variety of steels as determined by the nature and amount of alloying elements present as well as the heat treatment given to such steels.

The effects of austenitizing temperature and time were studied in particular detail in the 5 per cent chromium, 0.8 per cent carbon steel and considerable data were obtained on the carbides formed on isothermal transformation at various sub-critical temperatures for various periods of time when soaked for 1 hour at 1100°C. Extracted carbides generally appeared to be of the  $\text{Cr}_7\text{C}_3$  type, excepting in the lower chromium steel, where the carbide additionally showed the  $\text{Fe}_3\text{C}$  structure. A transitional iron carbide also appears to be formed at the beginning of the transformation at lower temperatures.

Work on three vanadium steels with vanadium to carbon ratio of 1:1, 1:5 and 1:15 was also taken up. With 3 per cent vanadium and 0.6 per cent and 0.2 per cent carbon respectively, and with 1 per cent vanadium and 1 per cent carbon, the carbide extracted from fully normalized specimens showed a face-centred cubic structure corresponding to vanadium carbide. Proper soaking temperature and time were also investigated as these are of considerably greater importance in these steels in view of the higher stability of the carbide.

Further isothermal transformation studies were continued on two chromium steels and a vanadium alloy steel. Transformation was carried out at temperatures in the range 400°-700°C. and for periods ranging from 1 minute to 10,000 minutes. The specimens after transformation were subjected to both metallographic as well as hardness examination and precipitates from these specimens were subsequently extracted for X-ray examination using the ultrasonic beam

bombardment method. The X-ray examination of the extracted carbides is being carried out.

An attempt is being made to develop the ultrasonic method of extraction of precipitates for the quantitative assessment of carbides in steel. A preliminary experiment was made on a high-carbon, high-chromium die steel, quenched from 950°C., with very satisfactory results. As the proportion of carbides can be more accurately estimated in normalized and quenched plain carbon hyper-eutectoid steels, quantitative standardization experiments were carried out with a 1.32 per cent carbon steel. In these studies, specimens of this steel were soaked at temperatures differing by 50°C. from 725°C. upwards, quenched in a 10 per cent brine solution kept at 18°C. and subsequently bombarded with ultrasonic beams for the extraction of carbide and its quantitative assessment.

### 37.0 Transformation of Austenite

This project has been taken up to elucidate certain aspects of the transformation characteristics of austenite and its retention. Some of the aspects are thermal stabilization of the austenite above and below the  $M_s$  temperature, the effect of interrupting the quench, the effect of isothermal hold and the effect of specific heat-treatment operations.

The general plan of work is to subject a suitably austenitized specimen to a specified heat-treatment operation, involving various amounts of stabilization over various ranges of temperature and quantitatively assessing the nature and extent of transformation. The latter is based upon hardness, metallographic and X-ray examination.

Work has been initially carried out on two chromium steels — one a 2.3 per cent carbon, 13.6 per cent chromium die steel, and the other a 1.1 per cent carbon, 2.83 per cent chromium tool steel. In both steels, after preliminary determination of the critical points and general transformation characteristics

as well as the  $M_s$  and  $M_f$  temperatures, suitably austenitized specimens were heat-treated in a variety of ways and austenite stability examined. A wide variety of quenching media, viz. water, 10 per cent sulphuric acid, 10 per cent sodium hydroxide, mercury, oil (all at 18°C.) and liquid air, with all but the last held at various higher temperatures, were used for the quenching and holding experiments. By varying the quenching temperatures and quenching media, it has been conclusively established that athermal stabilization of austenite takes place above the  $M_s$  temperature in both these steels. By interrupting the quenching at intermediate temperatures above the  $M_s$ , it has been further established that athermal stabilization is confined to a relatively narrow temperature range above the  $M_s$ . For the high-carbon, high-chromium die steel with an  $M_s$  of about 180°C., athermal stabilization was observed to occur over the range 185°-340°C. For the low-carbon, low-chromium steel, the corresponding  $M_s$  is about 93°C. and athermal stabilization has been observed to occur predominantly over the range 100°-300°C. A similar observation has also been made on a silicon manganese spring steel.

The quantitative assessment of the transformation of austenite by X-rays was taken up in detail. The technique used is similar to the one developed by Cohen and his colleagues and consists essentially in studying the intensity of the diffraction pattern obtained when a monochromatized beam of X-rays from a cobalt target is incident at an angle of about 60° on a plane polished surface of the specimen. Standardization work is in progress before quantitative assessment is carried out.

### 38.0 Lattice Parameters of Iron-chromium Alloys and Study of the Sigma Phase in Alloy Steels

Work has been taken up with a view to determine the nature and structure of the



sigma phase arising in alloy systems of the transition elements. The importance of this investigation can be appreciated from the fact that the presence of sigma phase generally renders steel unsuitable for fabrication and cold deformation.

The plan of work is to prepare from transition metals, of as high a degree of purity as available, alloys of various specified compositions and to examine their metallographic structures as well as lattice parameters.

On a thorough reassessment of the constructional features of an argon arc furnace proposed earlier to be constructed, it was decided to construct a smaller and less elaborate furnace in the first instance to gather necessary experience in the melting and preparation of the required high-purity alloys. Design and fabrication of the furnace parts were accordingly taken up in the laboratory workshop.

Simultaneously, attention was given to refining by the zone melting technique, commercial aluminium, as a preliminary to the construction of a suitable zone refining apparatus for adequate refining of such of the transition elements as cannot be obtained to requisite specifications. Attempts are being made to construct a suitable zone refining apparatus, using a small resistance type electric furnace, for producing the requisite molten zone.

### **39.0 Utilization of Vanadium-bearing Titaniferous Magnetite Deposits of Singhbhum and Mayurbhanj**

The vanadium-bearing deposits in India are located at Singhbhum and Mayurbhanj and contain about 1.5-2.5 per cent vanadium pentoxide with 10-16 per cent titanium dioxide. The conservative estimates put the reserves to about 20-22 million tons and investigations have been taken up for the utilization of these deposits for the production of vanadium pentoxide and subsequently ferro-vanadium. Experiments were

undertaken for the recovery of vanadium by salt roasting.

*Salt Roasting*—Laboratory studies on the recovery of vanadium pentoxide by salt roasting were completed. The factors studied for roasting include the effect of (a) particle size, (b) temperature, (c) different sodium salts, and (d) kinetics of roasting. The roasted mass was crushed and leached with hot water and vanadium pentoxide precipitated by adjusting the pH to 1.5-2. The effects of temperature and the pH on the recovery of vanadium pentoxide were studied and it was observed that an overall recovery of 60 per cent could be obtained. Based on the laboratory scale studies, full details of a pilot plant, capable of treating one ton of ore per day, were worked out and the plant is under installation. Studies were also carried out on the recovery of sodium sulphate from the liquor left after the precipitation of vanadium pentoxide.

### **40.0 Production of Iron-chromium-manganese-nitrogen Master Alloy**

This project has been taken up with a view to produce iron-chromium-manganese alloy of suitable composition to be utilized in the production of nickel-free stainless steel and other alloys.

Based on the studies on the production of iron-chromium-manganese alloys by aluminothermic reactions, a further quantity of one ton of alloy was produced during the year and 750 lb. of the alloy were nitrided.

### **41.0 Thermal Beneficiation of Low-grade Chrome Ores**

The large reserves of low-grade Indian chromites having Cr : Fe ratio less than 3 cannot be directly used for the production of high-grade ferro-chrome. The conventional ore-dressing methods fail to improve this ratio as FeO and Cr<sub>2</sub>O<sub>3</sub> are chemically bound in the chrome spinel. Attempts are being made to upgrade the ore by

preferential reduction of FeO and its subsequent removal by acid leaching.

A low-grade chrome ore from Tagadur area in Mysore, with an initial Cr : Fe ratio of 1.04 : 1, was taken up for investigation. The ore was crushed and different fractions studied for mineral constituents. The crushed ore was briquetted with varying quantities of the reducing agent and reduced from 1000° to 1300°C. The reduced briquettes were crushed and leached with hot sulphuric acid and then filtered. It was observed that reduction started at 1100°C. and the Cr : Fe ratio increased initially with rise in temperature and higher carbon content of the briquettes while chromium recovery decreased in the same order. At 1300°C., it was observed that higher coke content resulted in lowering the Cr : Fe ratio as well as chromium recovery. A Cr : Fe ratio of 3 : 1 was obtained when the reduction was carried out at 1300°C. for 3 hours with 10 per cent coke in the charge. Studies on the effects of other reducing agents like lamp black and activated charcoal and the particle size of chrome ore are being continued.

#### 41.1 Thermal Beneficiation of Low-grade Manganese Ores

The ferruginous manganese ores are not suitable for the production of standard grade ferro-manganese and could be upgraded by preferential reduction of iron, producing a manganese-rich slag. The iron obtained in the metallic form would be a useful by-product. The studies of manganese ore from Koraput, Orissa, assaying Mn, 38.9; Fe, 10.9 and P, 0.3 per cent, were continued in clay-bonded graphite crucibles and morgan salamander crucibles at 1350° and 1400°C. The slags obtained had Mn : Fe ratio ranging from 10 to 50, but recovery of manganese in the slag decreased with rise in temperature and longer reduction period. Considerable silica pick-up in the slag from the crucible was observed and it varied from 20 to 30 per cent

in the slag. The salamander crucibles withstood the corrosion better than clay-bonded crucibles, the silica pick-up was the same in both the cases.

A few experiments were performed to reduce the silica and alumina content of the slag (34.06 per cent SiO<sub>2</sub> and 2.68 per cent Al<sub>2</sub>O<sub>3</sub>) by roasting it with Na<sub>2</sub>CO<sub>3</sub> followed by leaching of the crushed calcine. It was found that a residue could be obtained assaying 15 per cent SiO<sub>2</sub> and traces of Al<sub>2</sub>O<sub>3</sub> by roasting the slag for 4 hours at 1050°C. followed by leaching the calcine with water at 90°C.

#### 42.0 Production of Low-carbon Ferro-chrome

The high-carbon ferro-chrome containing 6 per cent carbon is manufactured by the carbon reduction of chromite ores in arc furnace. Low-carbon ferro-chrome is not produced in India. Investigations have been taken up to produce low-carbon ferro-chrome containing up to 0.05-0.2 per cent carbon for use in alloy steels. It was earlier observed that by suitable melting of high-carbon ferro-chrome and ferro-silicon, the carbon content of the alloy could be considerably reduced and an alloy containing 40 per cent silicon was obtained with as low as 0.04 per cent carbon. The addition of molten ferro-silicon to molten ferro-chrome did not result in any violent reaction and carbon was eliminated. About 20 lb. of chrome-silicide was made by mixing together molten charges of high-carbon ferro-chrome analysing 59 per cent Cr and 6 per cent C with ferro-silicon assaying about 89 per cent silicon. The low-carbon chrome silicide product obtained analysed Cr, 30.55; Fe, 23.7; and Si, 44.15 per cent. A few experiments were carried out for desiliconization of the above product by treatment with chrome ore and limestone in an indirect arc rocking type of furnace lined with dead burnt magnesite. The kinetics of desiliconization and the effect of basicity were studied.

#### 42.1 Production of Carbon-free Ferro-chrome

There is today no production of carbon-free ferro-chrome in India. The project has been taken up with a view to prepare the material to meet the urgent requirements of the Metal and Steel Factory, Ministry of Defence, Ishapur.

Systematic studies on the production of ferro-chrome by aluminothermic reactions were undertaken and the effect of particle size, different energizers, rate of feeding, shape and size of the vessel and the effect of fluxes were studied. Based on these laboratory scale experiments, large-scale trials were conducted and ferro-chrome of the following composition was produced:

	%
Chromium	76-79
Iron	20-25
Aluminium	Less than 0.5
Silicon	1-2.5
Carbon	Traces

A total quantity of half a ton of ferro-chrome was made to rigid ordnance specifications and was sent to the Metal and Steel Factory, Ishapur, for industrial melts which have been highly successful. A further quantity of 250 lb. of carbon-free ferro-chrome for the ordnance plants has also been produced.

#### 43.0 Production of Magnesium from Dolomite

The investigation was taken up to produce magnesium by the silico-thermal reduction of dolomite. The pure magnesium so obtained will find varied applications in ordnance plants.

The reaction mechanism and the effect of various factors involved in the silico-thermic reduction process were studied and experiments were carried out in a small tubular electric furnace. The various factors studied include, effect of temperature, effect of addition of catalyst, and the effect of varied proportions of the reactants in the charge.

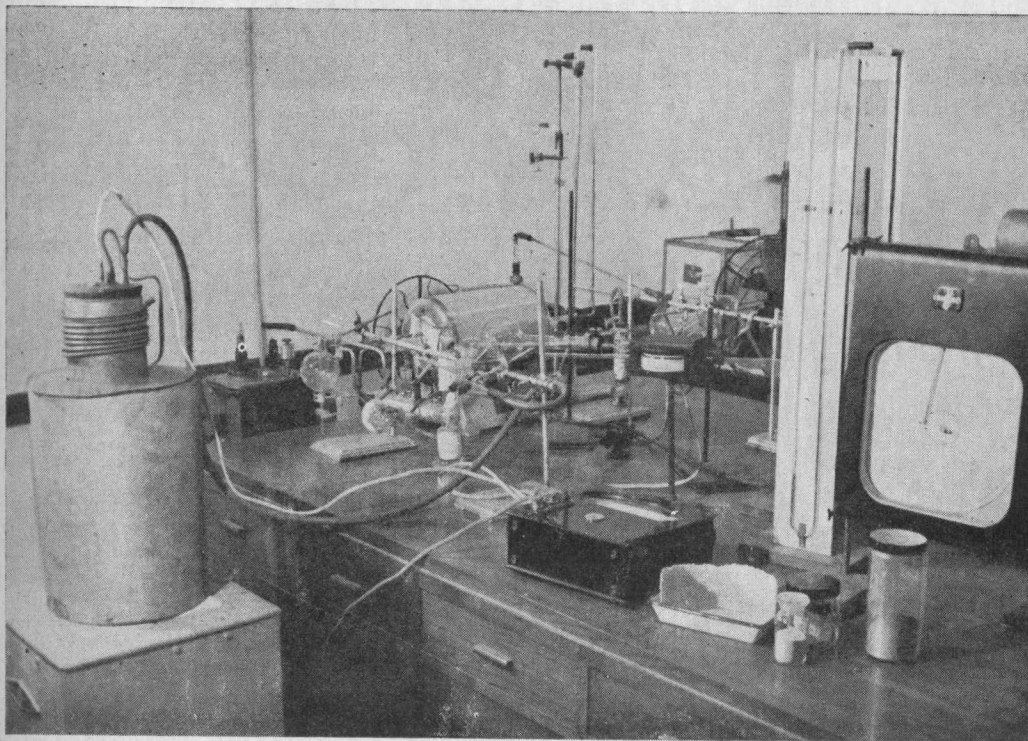
The reaction was observed to be sufficiently fast at 1100°C. under a vacuum easily attainable with the help of a commonly used rotary pump. Further studies on the effect of particle size of the reactants and reaction kinetics are under progress. The condensation characteristics of the magnesium vapour were studied in a specially designed split type fractionating condenser. The correct temperature of condensation for massive magnesium deposit was determined. It was possible to separate different volatile products such as manganese, silicon and alkalis in different temperature zone, away from the massive crystalline magnesium deposit. Based on laboratory scale experiments, a larger unit consisting of a 6 in. dia. vacuum retort was designed and fabricated. A few heats were carried out to produce 1 lb. of magnesium per batch. The metal so produced was analysed and found to contain only minute traces of impurities. It is now planned to increase the retort size so that larger quantities of magnesium may be produced per batch.

#### 44.0 Production of Magnesium Powder

This investigation was taken up to produce magnesium powder according to the specification of the High Explosives Factory at Kirkee for their urgent needs. Attempts were made to produce magnesium powder by the shock-cooling method. A water-cooled condenser was designed and fabricated. Initially magnesium turnings from an ingot were heated under vacuum. The magnesium vapours were condensed on the water-cooled surface at pressures of 1-3 mm. of mercury. The magnesium deposit obtained was quite brittle but was partly pyrophoric in nature and had to be crushed under an inert liquid.

The nature of imported magnesium powder was studied. The samples were sieve-analysed and their bulk densities determined. One pound of magnesium powder was made by milling the ingot turnings. The powders





EXPERIMENTAL SET-UP AT THE NATIONAL METALLURGICAL LABORATORY FOR THE PRODUCTION OF MAGNESIUM METAL

so obtained were suitably mixed to conform to rigid specifications laid down.

#### 45.0 Separation of Copper from Glanz Metal

Mixed white metal scraps after sweating leaves a hard alloy (glanz metal) analysing approximately Sn, 20; Sb, 10; and Cu, 30-40 per cent and the balance lead. This alloy as such cannot be commercially utilized due to the presence of large quantity of copper and it is proposed to separate copper from the alloy, so as to make residual alloy commercially useful for the manufacture of anti-friction and other similar bearing metals.

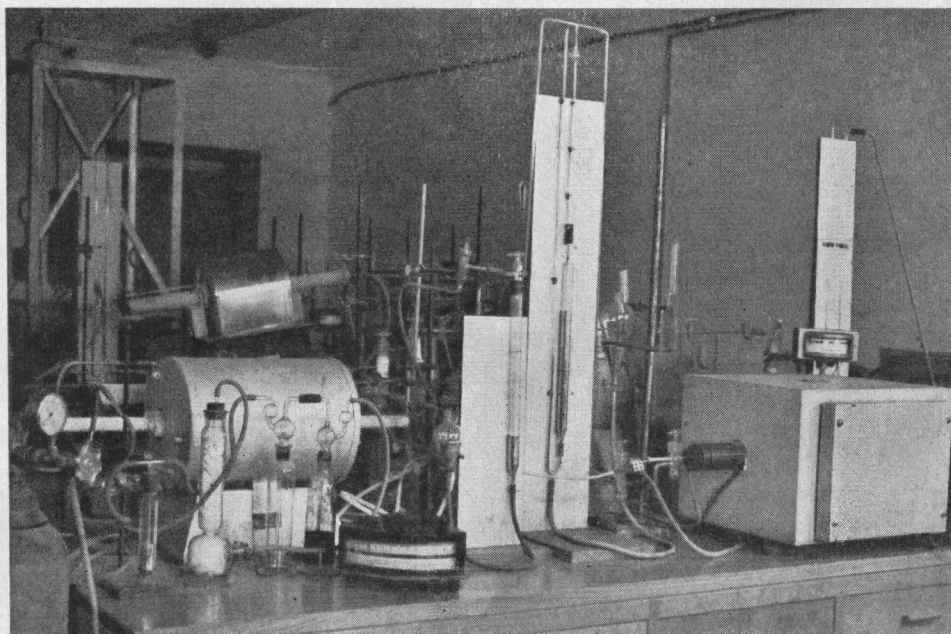
It was observed that on melting the alloy with pyrites, the copper is removed in the matte as sulphide. Systematic experiments were carried out using varying quantities of

pyrite and elemental sulphur at different temperatures for different periods of time. It was possible to eliminate 95.35 per cent of copper from the original alloy, but about 40 per cent lead, 43 per cent tin and 24 per cent antimony were also removed into the matte along with copper. The investigation is now completed and a full report thereon is being compiled.

#### 46.0 Recovery of Nickel from Nickeliferous Ore of Rajasthan

The nickeliferous ore of Rajasthan contains 0.25 per cent Ni and it is proposed to extract its nickel content economically.

Experiments were carried out to reduce the nickel oxide by hydrogen or carbon monoxide at elevated temperatures. It was observed that silica minerals were neither



SET-UP OF THE APPARATUS FOR THE DETERMINATION OF SOLUBILITY OF NITROGEN IN PLAIN CARBON AND ALLOY STEELS

reduced nor dissolved in ammoniacal solutions. Further experiments on the recovery of nickel were carried out using chlorination. The ore was crushed to  $-60$  mesh and the briquettes were chlorinated at elevated temperatures. The effect of temperature and the kinetics of chlorination were studied. It was observed that iron was also chlorinated along with nickel. Further experiments are under way to suppress the chlorination of iron.

#### 47.0 Solubility of Nitrogen in Plain Carbon and Alloy Steels

A study has been taken up with a view to determine the solubility of nitrogen in nickel-free austenitic chromium-nitrogen-manganese-copper stainless steel in solid and liquid state.

The assembly for the determination of the solubility of gases in metals at different temperatures in solid state was set up and the volume of the unit was determined. Certain modifications were carried out in the light of

the experience gained, as it was observed that the volume of the apparatus was not sufficient. Preliminary experiments carried out on the determination of solubility indicated that the absorption was very slow and was attributed to the formation of an oxide coating. The cylinder gas commercially available contained considerable amount of oxygen and the purification train is being modified to remove the last traces of oxygen. Initial experiments on the solubility of nitrogen will be carried out in 18:8 nickel-chromium stainless steels and the values checked with the data of other workers. The studies on the solid solubility of nitrogen in nickel-free austenitic stainless steel will be taken up thereafter.

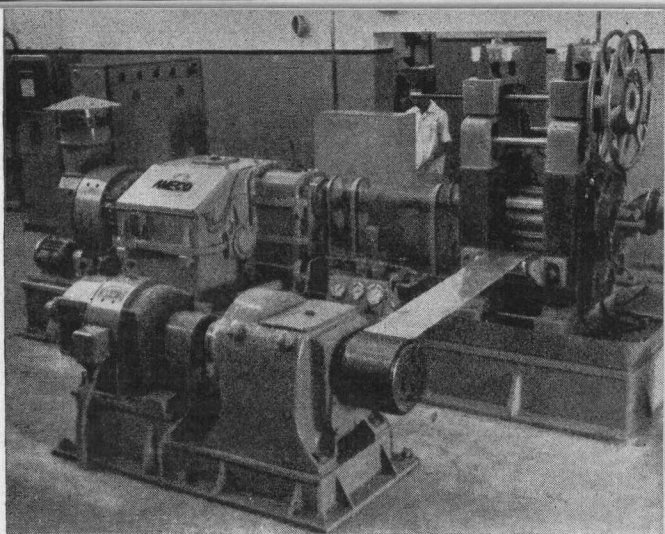
#### 48.0 Development of Low-alloy High-tensile Structural Steel

This is a broad-based investigation which has been taken up with the object of developing suitable low-alloy structural steels

possessing good mechanical properties, working characteristics, corrosion resistance and weldability. The main aim of the project is to find out suitable compositions for low-alloy high-tensile steel utilizing mainly indigenous alloying elements.

In the first phase, the work was concentrated in making laboratory scale heats of the existing low-alloy high-tensile steel with slight modification of the percentages of alloying elements. This was done with a view to standardize the conditions of the laboratory scale production of high-tensile steels. Main alloying elements of the first series were manganese, chromium, silicon and copper. From the test results of the series it was found that steels having manganese in the range of 1-1.5 per cent, chromium in the range of 0.6-1 per cent, silicon in the range of 0.3 per cent and copper in the range of 0.25-0.4 per cent possess good mechanical properties combined with good welding characteristics.

In the next series of work, additions of small percentage of vanadium and titanium were made to harden the ferrite as well as to improve the grain structure. Additions of aluminium and zirconium were also made with the idea of getting finer grain sizes. Carbon, in all cases, was limited to a maximum of 0.18 per cent so that the weldability of the steel was not impaired. In this series, basic composition was kept within the limit as stated above. Addition of vanadium of the order of 0.1-0.15 per cent was noticed to strengthen the steel considerably. Almost similar results were obtained with addition of titanium. From the test of the second series it was observed that a few compositions were giving quite encouraging results. A number of heats based on the second series of experiments were made with slight variations in the alloying percentage of manganese, chromium, silicon and copper. Most of the heats of the last group gave tensile strength of the order of 45 tons per sq. in. However, it was noticed that heats having 0.1-0.15 per cent titanium were, in some



THREE-HIGH REVERSING ROLLING MILL INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

cases, giving slightly less low temperature impact properties.

During the work, one heat was made with carbon in the same range but with much higher silicon and slightly higher manganese than the previous heats. The heat gave excellent forging and rolling characteristics as well as exceptionally high mechanical properties. The tensile strength was of the order of 90 tons per sq. in. in the rolled, forged, normalized and annealed condition. Although this could not be placed in the group of low alloy steel, as the total alloying content was of the order of 6 per cent, it may provide a very good constructional steel. Low percentage of carbon may make it a good carburizing steel. Further work is in progress.

#### **49.0 Development of Electrical Resistance Alloys for Heating Elements**

All electrical resistance heating elements are today imported in India. The more common types of these alloys, e.g. nichrome and kanthal, contain high percentage of nickel and cobalt neither of which is available from indigenous sources. The aim of this project is to develop substitute alloys with alloying elements available from indigenous resources



which can be used for domestic and industrial heating purposes.

The work was centred round iron-chromium-aluminium system due to indigenous availability of alloying elements. The alloying elements used, besides Cr and Al, were Mn, Si, Ti and Zr in various proportions. The last two were chiefly intended for refining grain size and checking grain growth at high temperatures. Some new alloying elements like Cu, Cu + N, B + N and P were tried, but the workability was found to be impaired by their additions. Too high a percentage of Si or Ti had deleterious effects. Mischmetal additions were found to be very beneficial. Work was also done on low-chromium, high-aluminium alloys with other suitable alloying additions.

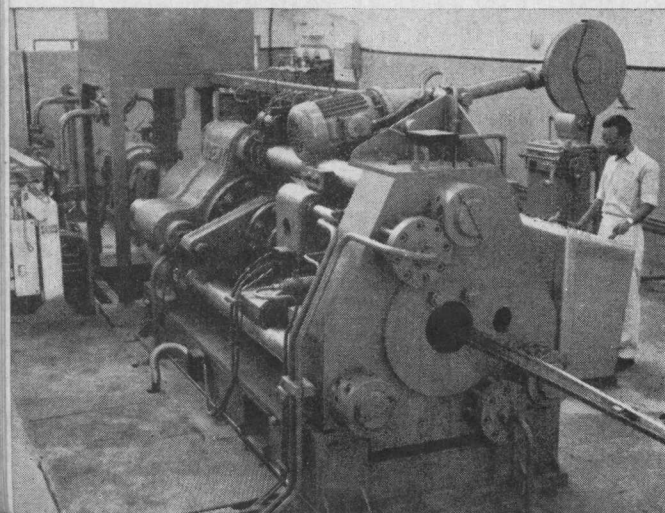
A large number of heats were prepared in the high-frequency furnace. Armco iron, low-carbon ferro-chrome, silicon-metal, electrolytic manganese, commercially pure aluminium, Zr-Al alloy, ferro-titanium, etc., were used as charge. In a few cases, a Cr-Al master alloy of suitable Cr: Al ratio was used as charge instead of ferro-chrome and aluminium with a view to minimize alumina content in the heat and ensure very low carbon content, both of which were found to affect workability adversely. The ingots made, in most cases, were of 4 lb. except in some of the heats which were made in 20 lb. H.F. furnace. Hot-forging was carried out at 1150°C. initially and at 800°C. finally so as

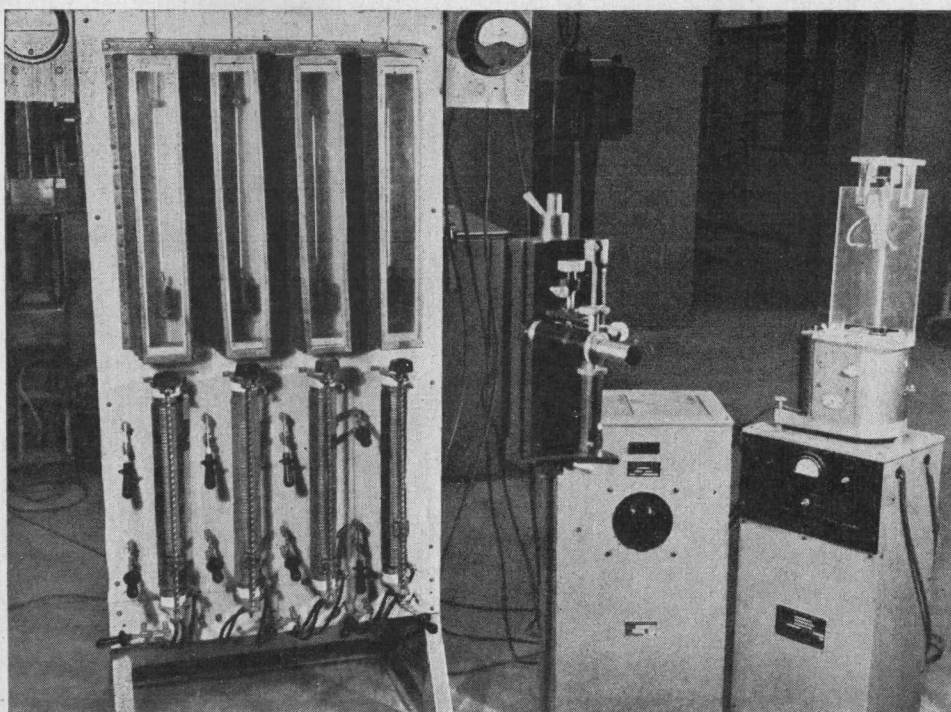
to refine the grain size. At intermediate stages during forging, surface defects were removed by machining. Bars were ground to round shape and cold-drawn. During cold-drawing experiments it was noticed that with the use of lime coating and soap solution as lubricant, the drawing was easy and smooth. Annealing at intermediate stages, after about 40-50 per cent reduction, was done at 700°C. for  $\frac{1}{2}$  hour. Wires were drawn even by double die reduction to 30 S.W.G. sizes. Mechanical and physical properties such as electrical resistivity and temperature coefficient of resistivity were accurately determined. Electrical resistivity of most of the heats were higher than that for nichrome. Rise of resistivity with temperature in high temperature ranges was also not appreciable. Some of the as-forged bars of  $\frac{3}{4}$  in. square section were hot-rolled to round section of  $\frac{3}{8}$  in. dia. size at the Metal and Steel Factory, Ishapur. Rolling was satisfactory. During wire drawing, snapping was noticed after 5 S.W.G. size. The causes are being studied. Later experiments showed that wire drawing by adopting suitable technique could be carried out without previous cold-rolling.

An apparatus for accelerated life test of heating elements based on A.S.T.M. specifications was designed and fabricated in the National Metallurgical Laboratory. An automatic "on-off" device for 2 minutes' duration was set up. Arrangements were also made for high temperature tensile testing of the alloys with the Hounsfield tensometer.

Electrical resistivity measurements of the alloys at elevated temperatures showed rapid rise in initial ranges of temperatures up to 500°C. after which the resistivity temperature curves flattened out. Constancy of resistivity at high temperature is a desirable feature of such alloys. Further work is being carried out to arrive at suitable compositions and to develop proper techniques for melting, casting and mechanical working of the ingots to finished fine-gauge wires. Due to inherent brittleness and grain growth tendency of the alloys, extreme care and control of all the

500-TON EXTRUSION PRESS INSTALLED AT THE  
NATIONAL METALLURGICAL LABORATORY





APPARATUS FABRICATED AT THE NATIONAL METALLURGICAL LABORATORY FOR THE DETERMINATION OF ACCELERATED LIFE TEST OF HEATING ELEMENTS

variables are necessary. Work towards this direction is in active progress.

### 50.0 Development of Technique for Production of Bimetals

Bimetals are extensively used in various kinds of temperature-controlling devices, electrical contact switches, etc. The manufacturing methods for bimetal are closely guarded secrets. In view of the interest shown by the Indian industries in the production of bimetal, the project has been undertaken with a view to develop a suitable technique for its production.

The selection of low and high expansive components is primarily dictated by the nature of use of the bimetal, the range of operating temperature, the sensitivity expected and the current rating. In general, the component alloys should have widely differ-

ent coefficient of thermal expansion, high Young's modulus and high proportional limit. For high temperature use, the bimetal should have good elastic properties at high temperature. bimetal used in the electric circuit-breaker should have, in addition to above, high electrical resistance also. For low expansion component, Fe-Ni alloys containing Ni from 36 to 46 per cent are universally used. For high expansion component austenitic stainless steel, Fe-Ni-Mn alloy, Mn-Cu-Ni alloy and brass are used.

Various methods were employed for bonding bimetal. The technique adopted is the direct hot-rolling method. Two slabs of requisite thickness of the alloys, one from high expansion group and the other from low expansion group, were welded along the periphery; after the bonding surfaces were properly cleaned. The surfaces were made fairly flat and pressed tightly together during

welding in order to avoid excessive oxidation of the bonding surfaces. Bonding was effected by hot-rolling the sheets at a proper temperature. After hot-rolling, the sheets were soaked for about one hour followed by heavy cold work and successive annealing at a lower temperature. The sheets were finished by giving about 30 per cent cold reduction so as to have enhanced elastic properties followed by a stress relief annealing at a temperature of 300°C. for a very long period. Work is in progress to improve the casting and working techniques of Mn-Cu-Ni alloys and improve the technique for bonding them with invar. Trials are also being made to develop a suitable technique to bond brass with invar.

### 51.0 Development of Controlled Friction Material

The investigation was taken up at the instance of the Ministry of Railways to develop controlled friction materials having a coefficient of friction not exceeding 0.16 when put into service.

The experimental investigations conducted earlier showed that the combination of grey cast iron and processed fabric liner (drilling holes  $\frac{1}{16}$  in. diam. and  $\frac{1}{16}$  in. deep on the surface and treated in molten stearic acid) produced, even after long service, desired range of coefficient of friction. Based on these results a service trial of the processed fabric liner was carried out by the Railway Research Centre, Lucknow. Processing of fabric liners was done according to experimental findings. But instead of grey cast iron, phosphor bronze was used as mating material in service. Service tests were carried out in locomotives and high coefficient of friction was found in service. The causes of this unusual behaviour are under investigation.

### 52.0 Development of Clad Metals

The process of cladding aluminium on mild steel and the optimum conditions required

for obtaining good bond strength have been described in earlier reports. Work has been carried out on the development of bonding technique for aluminium base bearing alloys which are bonded to other suitable backing alloys for use as bimetallic bearing. In this connection, attempts are being made, in the first instance, to make a suitable aluminium base bearing material with improved bearing properties. The heats of the compositions, (a) Sb, 4; Pb, 4 per cent, and balance Al, and (b) Sb, 1; Pb, 1.1; Mg, 0.5 per cent, and balance aluminium, were cast in steel moulds after melting in the electric furnace.

Using the same technique developed for bonding aluminium with mild steel, the bimetallic bearing strip was bonded to mild steel by the following process:

The bimetallic strips were first made by rollings in composite block of the alloys (a) and (b) at 400°C., followed by annealing for 2 hours at 450°C. Similar type of packs as described in the earlier report were made with the aluminium bimetallic strip on one side of the base metal, the former being folded on the latter on all sides. Optimum conditions of working as obtained in the earlier experiments (5 per cent cold reduction, 45 per cent hot reduction, 4 hours' annealing at 500°C.) were maintained. The rolling temperature was kept between 450° and 500°C. Metallographic observation of the bond zone showed an intermetallic diffusion. Further work is in progress.

### 53.0 Photo-elastic Stress Analysis

Study of distribution of stress in engineering components is necessary from the point of view of understanding the service behaviour of the components as well as for improving the design. With this view the work of photo-elastic stress distribution was undertaken.

Key-hole type of notches are used in various machine parts. Quite a number of service failures are known to have originated



from the root of key-hole notches. With a view to understand the behaviour of various types of key-hole notches, analysis of stress by photo-elastic method is being made with notches of various geometry having different combinations of depth and width in the centre of rectangular beam of photo-elastic material. The beams are being loaded in

pure bending with the notch in the compression side. Bending moment of known intensity is applied to the beams. The shear stress along the vertical line of symmetry for various notches is being compared from the isochromatic pattern. The stress concentration factor in all cases is also being determined.

## PILOT PLANTS

### 54.0 Low-shaft Furnace Pilot Plant Project

This project has been started with the object of conducting comprehensive research investigations on the production of foundry grades of pig iron from Indian raw materials such as soft iron ore, iron ore fines and beneficiated magnetite iron ore employing essentially non-metallurgical non-coking fuels such as high-ash coal or carbonized lignite and such fuels plentiful supplies of which exist in India but are unsuitable for utilization in conventional iron blast furnaces. The production of standard and exportable grade of ferromanganese in low-shaft furnace is also envisaged. The results of these comprehensive investigations will be of immense value for the establishment of regional iron and steel production centres in different parts of India.

The operation of the low-shaft furnace was started early in 1959. The period under review had been one of ceaseless activity, not only in bringing the constituent units of the plant into operation and co-ordinating their integrated working but also in training the staff in the round-the-clock operations of a full-scale pilot plant of this magnitude — the first of its kind in India. The working and operational difficulties and hazards of a low-shaft furnace do not follow the operation of a highly mechanized standard iron blast furnace and as such unforeseen operational and maintenance difficulties were encountered which were successfully overcome. As different grades of raw materials are to be tried, the operational problem of such a plant can never be wholly predetermined and have to be dealt with in course of operation itself.

The programme of the operation of low-shaft furnace has been divided into the following phases:

#### *Phase I*

(i) Smelting of iron ore from Orissa and Bihar, with non-coking coals from Raniganj and Disergarh coalfields — either in bedded charge or by smelting of self-fluxing briquettes.

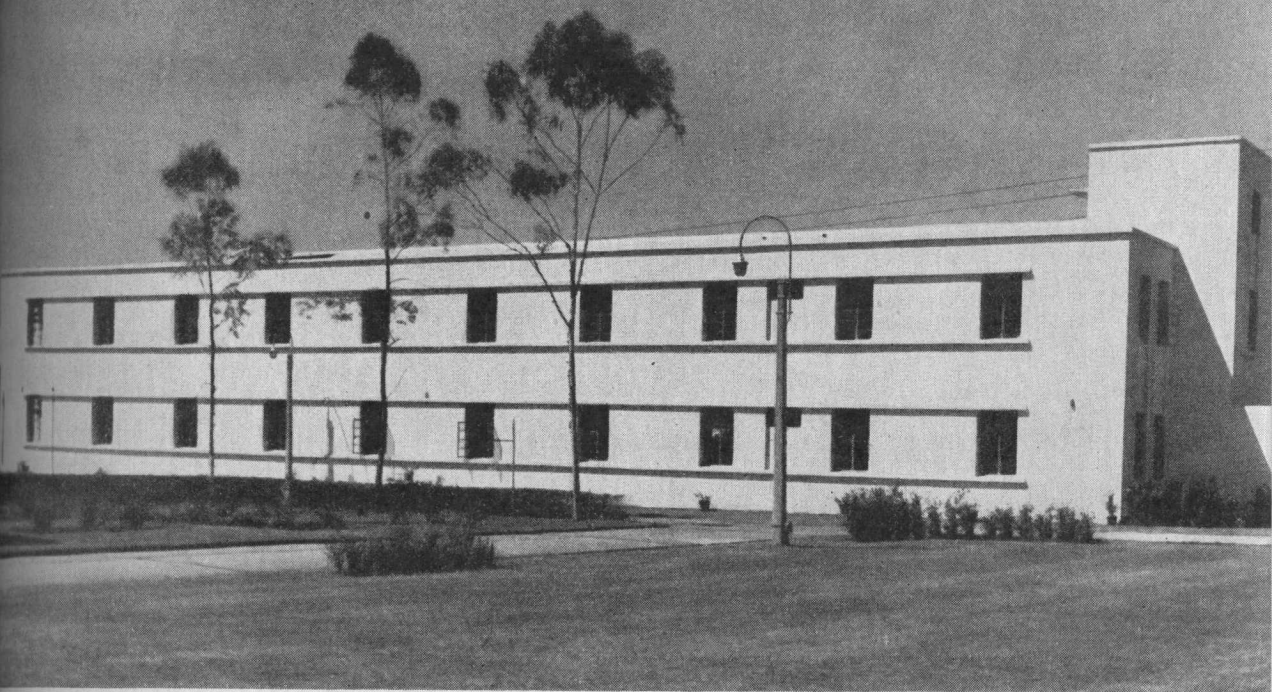
(ii) Smelting of iron ore from Chanda District with non-coking coals from Ballarpur, Kampti and Wardha Valley, Bombay State.

(iii) Smelting of iron ore from Morinza near Jaipur and Udaipur, Rajasthan, initially with non-coking coal and then with Palana lignite [ vide Phase III, (i) ].

(iv) Smelting of iron ore from Siddhi in U.P. with non-coking coal from Singrauli ( Rewa belt ), etc.



PROF. M. S. THACKER, DIRECTOR-GENERAL, SCIENTIFIC & INDUSTRIAL RESEARCH, OPENING THE BAY HOUSING THE EQUIPMENT RECEIVED FROM GOVERNMENT OF U.K. UNDER COLOMBO PLAN



THE NEW PILOT PLANT TECHNOLOGICAL BAY AT THE NATIONAL METALLURGICAL LABORATORY WHICH WAS OPENED BY PROF. M. S. THACKER, DIRECTOR-GENERAL, SCIENTIFIC & INDUSTRIAL RESEARCH, ON 15TH FEBRUARY 1960

(v) Smelting of iron ore from Chhapra, Antribeharipur in Mohindragarh, Punjab, with non-coking coals.

(vi) Smelting of iron ore from Anantpur, Warangal, etc., with non-coking coals from Kothaguddium, Yallandu, etc., in Andhra Pradesh.

(vii) Smelting of iron ore from Katni in M.P. with non-coking coals from adjacent coalfields (Kanhana and Panch Valley).

#### *Phase II*

Smelting of self-fluxing briquettes made from iron ore fines, limestone and non-coking slack coals.

#### *Phase III*

(i) Utilization of carbonized lignites from Neyveli and Palana.

(ii) Utilization of beneficiated Salem magnetite.

#### *Phase IV*

Fuel economy — Injection of top gas through the tuyeres, sintered burden, oxygen enrichment of blast, etc.

#### *Phase V*

Production of ferro-alloys, viz. ferro-manganese and ferro-chrome.

Currently investigations under Phases I (i) and II are being conducted and Phase I (ii) will commence shortly.

In the first two campaigns, the furnace was operated with lumpy iron ore, limestone and nut-coke mainly to initiate co-ordinated function of all ancillary equipment as an iron-producing unit. In the third campaign, the fuel burden was gradually changed to 100 per cent non-coking coal, which is the main objective of the smelting trials in this pilot plant. The chemical analyses of raw materials employed were as shown in Table 3.



TABLE 3

Iron Ore						
LOCATION	Fe, %	SiO <sub>2</sub> , %	Al <sub>2</sub> O <sub>3</sub> , %	MgO, %	S, %	P, %
Barbil ( Orissa )	57	4	4.5	0.5	0.3	0.04
Non-coking Coal						
	H <sub>2</sub> O, %	ASH, %	V.M., %	F.C., %		
Raniganj field						
Samla Colliery	4.50	14.00	34.00	47.50		
Ghusick Muslia Colliery	4.50	25.60	31.10	38.80		
Jaipuria Colliery	3.50	20.50	35.00	41.00		
Jambad Colliery	3.48	20.48	34.94	41.00		
Sirka	2.40	22.90	30.00	44.62		
Limestone						
	CaO, %		MgO, %	SiO <sub>2</sub> , %		
Birmitrapur, ( M.P. )	48.85		5.94	5.02		

Although no difficulty was experienced during smelting with coke, its replacement by coal introduced serious operational difficulties due to frequent bridge formation causing hanging in the furnace stack and irregular descent of the burden. Very often the furnace explosion doors opened out to release the excessive pressure built up due to bridge formation. Besides, a higher amount of non-coking coal was initially necessary to generate adequate smelting capacity and temperature. The increase in fuel, coupled with the high ash content of non-coking coals, resulted in high slag volume. The operation was exceedingly susceptible to minor burden changes and attempts to reduce the fuel consumption often caused chilling of the hearth.

The furnace was operated on the basis of the following operational conditions:

Temperature of the  
hot blast 550°-600°C.

Temperature of top  
gas 350°-450°C.

Volume of air blast 2000-2500 Nm<sup>3</sup>/hr.

Pressure of blast 2000-2500 mm./WC

In the fourth campaign, the non-coking coals from Jaipuria, Jambad, Samla and Sirka collieries of the Raniganj field were employed for smelting. All these coals are characterized by high volatile matter and high ash contents — the latter chiefly containing over 50 per cent SiO<sub>2</sub> and 25 per cent Al<sub>2</sub>O<sub>3</sub>. Haematite iron ore from Barbil in Orissa ( Fe, 57.0; SiO<sub>2</sub>, 4.6; Al<sub>2</sub>O<sub>3</sub>, 3.2; S, 0.02; and P, 0.03 per cent ) and limestone from Bisra ( CaO, 44.8; MgO, 5.9; and SiO<sub>2</sub>, 5 per cent ) were employed in these investigations. The smelting operation was controlled by suitable adjustments of tuyere diameter through fixing reducers with consequent change in blast pressure ( 1800-2000 mm./WC ), blast volume ( 1500-2000 Nm<sup>3</sup>/hr. ) and blast temperature ( 550°-590°C. ). The top gas temperature under these conditions varied between 400° and 500°C. The top gas temperature was of the order of 500°-600°C. when the furnace was operated exclusively with coke. After extensive trials, satisfactory standard grade foundry pig iron analysing ( C, 2.8-3.3; Si, 3.0-3.3 and S, 0.03-0.06 per cent ) could be made only when the rate of drive of the furnace was slowed down by the employment of 45 mm. dia. tuyeres resulting in a production of pig iron of only 3 tons per day. Under these conditions, the abnormally high fuel rate in terms of fixed carbon was 4.0 tons per ton of pig iron. The slag under these conditions analysed 40 per cent CaO and 32 per cent SiO<sub>2</sub> containing very little iron. The high consumption of fuel was uneconomical and was considerably reduced in subsequent trials, as reported later.

For the operation of the gas-cleaning plant the rate of flow of gas was regulated by fixing a butterfly valve. It was concluded that despite high consumption certain grades of wholly non-coking coal, which are non-friable in nature and do not possess binding

qualities on carbonization, can be employed directly in the smelting of iron in a low-shaft furnace.

Along with the operation of the furnace, extensive trials were conducted to make suitable self-fluxing briquettes for iron production. For this purpose, a large number of modifications were incorporated in the briquetting plant as well as in the smelting procedures. The crushed raw materials (0-10 mm. ) were briquetted with addition of 5 per cent coal-tar pitch and 5 per cent sulphite lye as binders. The suitability of these briquettes was determined in the briquette testing unit and it generally depended on the coking index of the coal. The briquettes contained 63.6 per cent coal, 18.2 per cent iron ore and 18.2 per cent limestone.

For smelting the amount of briquettes in the normal burden consisting of lumpy iron ore, limestone and nut-coke was gradually increased. However, the smelting was associated with great difficulties primarily due to the disintegration of briquettes leading to the accumulation of dust in the furnace.

It has been mentioned earlier that the consumption of fuel was extremely high. In order to economize it, trials were conducted in the fifth campaign with the following objectives in view:

- (i) variation of the fuel consumption with the particle size of raw materials, particularly iron ore and limestone;
- (ii) optimum production in relation to the rate of blowing;
- (iii) control of the grade of iron produced;
- (iv) influence of increasing quantities of self-fluxing briquettes on the furnace operation; and
- (v) operation of the furnace exclusively with self-fluxing briquettes.

Bearing the objectives (i), (ii) and (iii) as mentioned above, the furnace was operated with non-coking coals. The analyses of raw materials were as shown in Table 4.

T A B L E 4				
Non-coking Coals				
LOCATION	H <sub>2</sub> O, %	ASH, %	V.M., %	F.C., %
Sirka ( Bokharo- Ramgarh-Ka- ranpura field )	2.4	22.9	30	44.62
Saunda ( Rani- ganj field )	7.7	9.8	31	51.50
Iron Ore				
	Fe, %	SiO <sub>2</sub> , %	Al <sub>2</sub> O <sub>3</sub> , %	
Barbil ( Orissa )	58.0	4.0	7.0	
Noamundi	63.0	3.5	6.0	
Limestone				
QUALITY	CaO, %	MgO, %	SiO <sub>2</sub> , %	
Limestone ( chips )	44.80	3.27	6.96	
Limestone ( lumps )	43.85	6.88	6.00	
Dolomite		Nut-coke		
	%		%	
CaO	30.20	H <sub>2</sub> O	0.80	
MgO	19.10	V.M.	0.96	
SiO <sub>2</sub>	1.66	Ash	23.64	
Al <sub>2</sub> O <sub>3</sub>	2.04	F.C.	74.60	

The furnace was blown with 60 mm. dia. tuyeres with a hot blast volume of 1900-2450 Nm<sup>3</sup>/hr., pressure of 1900-2600 mm./WC temperature of 560°-600°C. The top gas temperature varied between 390° and 570°C. and it was determined that it can be controlled by proper adjustment of particle size of the burden. Under these conditions a daily production of 4-8 tons was obtained, the fuel consumption being 2.9 tons of fixed carbon/ton of pig iron. The pig iron analysed:

	%
C	2.5-3.5
Si	2.5-5.0
S	0.02-0.10
Mn	0.3
P	0.6

The slag analysed:

	%
CaO	38.44
SiO <sub>2</sub>	30.33
FeO	0.9-1.5
Al <sub>2</sub> O <sub>3</sub>	26

and the slag volume was 1.38 tons/ton of pig iron. The top gas analysed:

	%
CO	30.0
CO <sub>2</sub>	3.5-5.0
H <sub>2</sub>	1.0
CH <sub>4</sub>	3.5
N <sub>2</sub>	65

The addition of small amount of dolomite ( 13 per cent of the flux addition ) was beneficial in favouring the carbon pick-up in the pig iron and improving fluidity of the slag.

In order to assess the maximum daily output of the furnace, the furnace was operated with the maximum tuyere diameter of 100 mm. admitting a blast volume of 4000-4100 Nm<sup>3</sup>/hr. at a pressure of 2100-2200 mm./WG. The raw materials consisted of dust ore, limestone chips, nut-coke and non-coking coal from Sirka Colliery. Under these conditions the operation of the furnace become uncontrollable as the explosion doors opened out at frequent intervals, presumably due to use of fine ground raw material. The calculated rate of production was 14.3 tons/day. With higher output, the fuel consumption was further reduced to 2.2 tons of fixed carbon and a slag volume of 1.24 tons per ton of iron made.

In order to reduce the dust generated from the briquette, factors affecting its quality were ascertained, but in the absence of a drying plant, the amount of moisture in the raw materials which exercises a profound influence on the quality could not be controlled. It would, therefore, not be possible to make briquettes during the monsoon, which will affect the running of the plant in this season.

For smelting trials the amount of the briquette in the burden was gradually increased

to 50 per cent, the other ingredients being lumpy iron ore, limestone and nut-coke. The furnace was blown with tuyere diameter of 60 mm. at a blast volume of 1900-2300 Nm<sup>3</sup>/hr. at a pressure of 2100-2400 mm./WG, for about 14 hr. The daily production amounted to 11.7 tons. The pig iron analysed:

	%
C	3.0-3.4
Si	2.0-4.8
S	0.06-0.10
P	0.7

The slag analysed:

	%
CaO	39.6-41.0
SiO <sub>2</sub>	32-34
FeO	1.0
Al <sub>2</sub> O <sub>3</sub>	22

The fuel ratio was 2.5 tons and the slag volume was 1.30 tons per ton of pig iron. The gas composition was 28 per cent CO, 4 per cent CO<sub>2</sub>, 2.0 per cent H<sub>2</sub> and 3.5 per cent CH<sub>4</sub>. Compared to the use of lumpy raw materials a higher amount of dust was generated.

The proportion of briquettes was then increased to 75 per cent. The blast volume and pressure were 2400-2500 Nm<sup>3</sup>/hr. and 2300-2500 mm./WG respectively. There was no change in the slag volume, fuel rate per ton of iron made and the output. Although the gas-cleaning plant was also in continuous operation, the fluctuation of the top gas pressure demanded continuous adjustment at the butterfly valve over the water seal and at the valve operating the final gas burner. It may be observed that increasing quantity of briquettes necessitated higher blast pressure which was due to the accumulation of dust in the furnace.

The last campaign was followed by two campaigns to operate the furnace exclusively with self-fluxing briquettes. While the smelting operation with 50-75 per cent briquettes and simultaneous running of the gas-clean-



ing plant presented no insurmountable odds, the attempts for smelting briquettes exclusively presented several difficulties. Although at the beginning no fundamental alterations of the operational characteristics were needed or noticeable, the blowing became progressively difficult due to accumulation of dust on top of the furnace. A large amount of dust was generated as was seen by the collection removed from the dust-catcher. The fine particles of dust were, however, not carried over and created the difficulties for blowing. The hanging and bridge formation at the top of the furnace did not permit regular descent of the burden. This resulted in lack of combustion in the hearth zone and sudden breakages of the bridge led to the descent of inadequately carbonized briquettes to the hearth area, thereby leading to the development of (a) insufficient heat for smelting, and (b) high top gas temperature. In consequence thereof the briquettes had to be charged very rapidly which again did not afford due time for carbonization of the briquettes and giving the latter due strength, etc. The sudden drop and poor strength of briquettes again contributed to the generation of dust. The trials, at the initial stages had to be discontinued as the penetration of the blast was affected. These difficulties have since been more or less fully overcome, which will be reported later.

Further work is in progress.

## 55.0 Semi-pilot Plant Production of Electrolytic Manganese Metal

Work was continued on the semi-pilot plant scale production of electrolytic manganese metal from low-grade manganese ore by the method developed and patented by National Metallurgical Laboratory. The manganese metal thus produced finds considerable application in the development of nickel-free stainless steel, nickel-free coinage alloy, substitute brass, etc., and many other alloys.

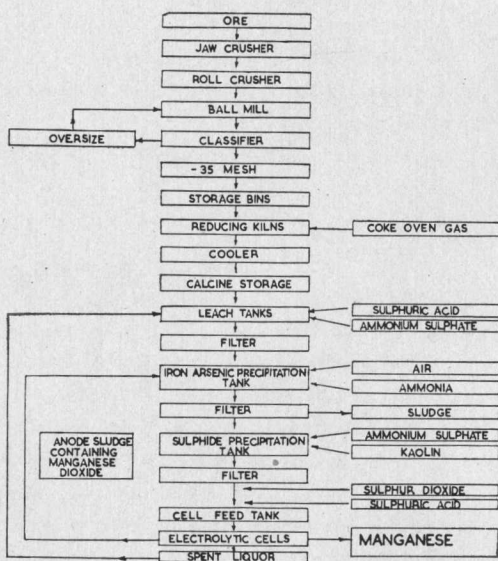
One 1000 amp. cell was fabricated and put into actual running. It produced 32 lb. of metal in 24 hr. and possessed the following improved features:

1. Better current efficiency;
2. Easier control of pH and temperature;
3. Uniformity of catholyte composition throughout the cell; and
4. Easier overhauling of the cell.

More than 1000 lb. of manganese metal was produced and supplied for experiments on coinage alloy and other research projects of the National Metallurgical Laboratory. Work was also continued on the installation of the 100 lb./day manganese plant. The design and layout of the plant equipment were complete. Almost all the major equipment excepting a few were received and the installation work is proceeding in full swing.

## 55.1 Semi-pilot Plant Production of Electrolytic Manganese Dioxide

The project has been taken up with a view to produce high purity manganese dioxide



FLWSHEET FOR PRODUCTION OF ELECTROLYTIC MANGANESE

from low-grade manganese ore, suitable for use in dry batteries. Laboratory scale experiments were successfully concluded and the product is now produced on a semi-pilot plant scale set up by the process developed and patented at the National Metallurgical Laboratory. Sufficient quantity of the material was produced to meet the requirement for testing experiments being carried out at the laboratory. A sample of 15 lb. was sent to an American firm for carrying out battery activity tests who have spoken very highly about the quality of the product.

The design and layout of the plant equipment for the 100 lb./day plant was completed and installation work is fast progressing.

### 55.2 Battery Testing of Electrolytic Manganese Dioxide

More than 100 dry cells (D type) were assembled according to the specification and tested. Different types of manganese dioxide including the N.M.L. produced manganese dioxide were used. The tests performed on the cells were:

1. 4 ohm intermittent tests after 10 days of assembly;
2. 4 ohm intermittent test after 6 months of storage on shelf;
3. continuous discharge test after 10 days of assembly; and
4. continuous discharge test after 6 months of storage on shelf.

The voltage and current of cells were regularly recorded at every stage during each test. The results obtained show the following salient points:

1. N.M.L. electrolytic manganese dioxide is a very satisfactory depolarizer for dry cells and gave performances comparable to those obtained from other sources.
2. Ampere hour output of cells obtained from cells made from N.M.L. electro-

lytic manganese dioxide is far superior to ampere hour output from similar cells made from natural ore in 4 ohm intermittent test.

3. Ampere hour output of cells obtained from cells made from N.M.L. electrolytic manganese dioxide is far superior to ampere hour output of similar cells made from natural ore in continuous discharge test.
4. The test mentioned as above on 6 months old cells also gave similar results.

### 56.0 Semi-Pilot Plant Studies on Beneficiation of Low-grade Ferruginous Manganese Ore from Koira, Orissa

The project was taken up on behalf of Messrs Orissa Manganese and Mineral (P) Ltd. to beneficiate the low-grade manganese ore deposits of their mines and to suggest suitable flow-sheet and machineries, etc., for the installation of a beneficiation plant by the firm.

About 8 tons of a low-grade ferruginous manganese ore from Koira, Orissa, were received which assayed Mn, 32.28; Fe, 22.06;  $\text{SiO}_2$ , 3.9;  $\text{Al}_2\text{O}_3$ , 5.5; P, 0.09; and BaO, 0.52 per cent. Psilomelane was the chief manganese mineral present followed by cryptomelane, pyrolusite and magnesite while goethite constituted the chief gangue mineral with minor amounts of quartz. Fair liberation of manganese minerals occurred at about 20 mesh.

Batch magnetizing reduction roast was carried out with -3 mesh sample and the reduced material was subjected to wet magnetic separation at different intensities at -3, -6, -10, -20, -28 and -35 mesh sizes. The results indicated that a good grade of manganese concentrate could be obtained with a recovery of 63.0 per cent Mn. Finer grinding did not appreciably improve the recovery. Based on the results obtained in the batch test, a large-scale reduction roast treatment was carried out with half a ton

of  $-1\frac{1}{2}$  in. ore in the vertical reduction furnace at the rate of 125 lb. of ore per hour. Magnetic separation of the reduced ore at  $-10$  mesh produced a concentrate, assaying Mn, 49.66; and Fe, 7.78 per cent with a recovery of about 60 per cent Mn. A continuous reduction roast of about 7 tons of  $1\frac{1}{2}$  in. ore was carried out successfully in the vertical reduction furnace at the rate of about 1 ton of ore per day. The reduced ore had been crushed to  $-10$  mesh and a large-scale magnetic separation is in progress to obtain a concentrate satisfying a Mn/Fe ratio of 7:1.

### 56.1 Semi-pilot Plant Studies on the Beneficiation of Low-grade Siliceous Manganese Ores from Orahuri Mines, Koira, Orissa

The study pertains to another sample of low-grade manganese ore sent by Messrs Orissa Manganese and Mineral Ltd. from

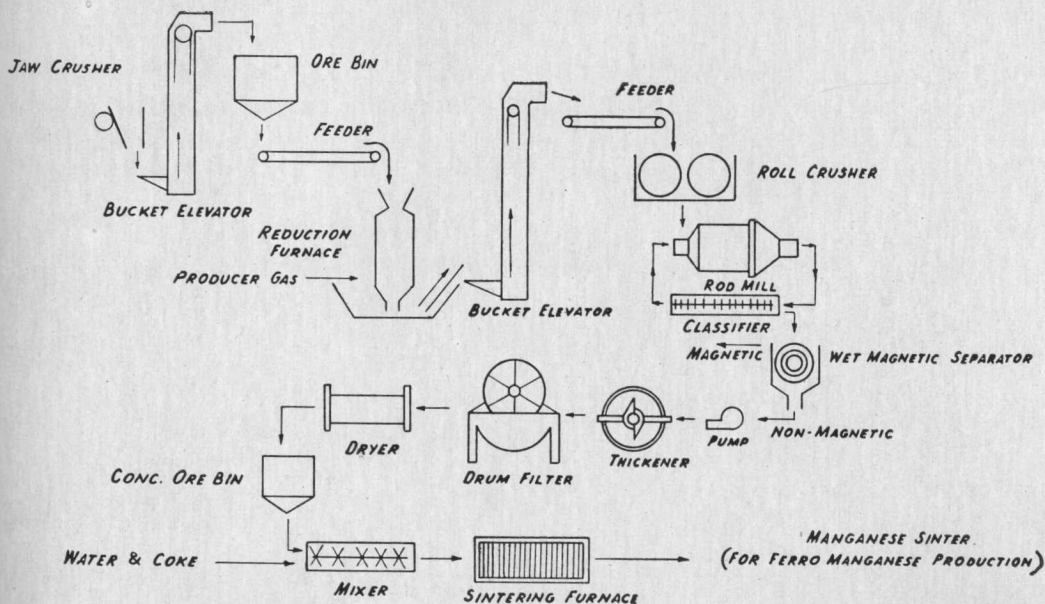
their Orahuri mine for beneficiation purpose.

The sample as received assayed Mn, 36.7;  $\text{SiO}_2$ , 31.05; Fe, 3.0;  $\text{Al}_2\text{O}_3$ , 2.5; and P, 0.04 per cent. High intensity magnetic separation at  $-20$  mesh and  $-35$  mesh yielded concentrates, assaying 52.7 and 52.9 per cent Mn with manganese recoveries of 79.4 and 72.2 per cent respectively.

### 57.0 Pilot Plant for the Treatment of Low-grade Manganese, Chrome and other Ores by Ore-dressing Methods

A pilot plant of  $\frac{1}{2}$  ton capacity per hour is being set up for treatment of manganese, chrome and other similar ores by ore-dressing methods.

The pilot plant is designed on the lines of a custom mill to treat different categories of manganese ores, chrome ores, etc. The plant is equipped with jaw and roll crushers for



A GENERAL FLOWSHEET FOR THE BENEFICIATION OF LOW-GRADE FERRUGINOUS MANGANESE ORE



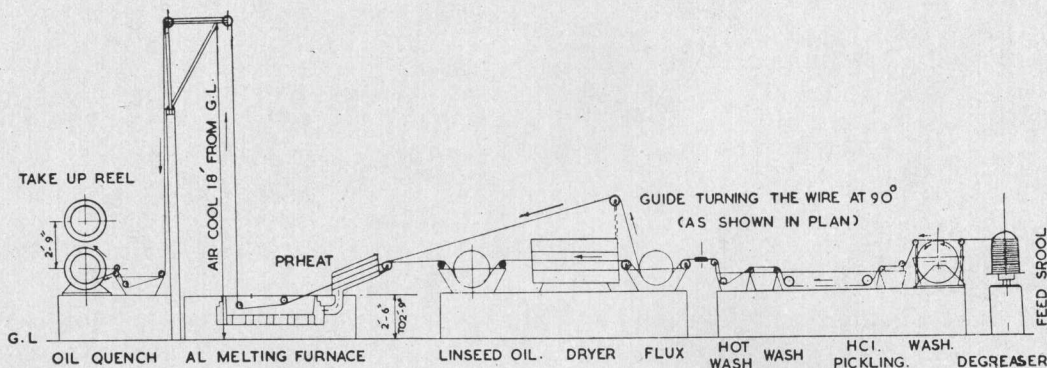
crushing, ball mill in close circuit with classifier for grinding, vibrating screens for screening, a vertical reduction furnace and a producer gas unit for carrying out reduction roast trials, dry magnetic separator, electrostatic separator, jig, shaking tables, gravel washer, thickener, filter, dryer, briquetting press with mixer, briquette hardening furnace, sintering hearth and auxiliary equipment like belt conveyers, bucket elevators, feeders, ore bins, etc. The flowsheet will be arranged according to the ore under investigation.

The raw ferruginous manganese ore 4-5 in. in size will be delivered into a platform from where it will be fed into a jaw crusher by means of a vibrating feeder. The jaw crusher discharge 1-1½ in. size will be stored in a bin. The crushed ore will then be elevated to the top of a vertical reduction furnace or a rotary furnace and discharged into the hopper of the furnace. Reducing gas from a producer gas unit entering the reduction furnace will reduce the iron oxides of the descending charge of ore in the furnace to magnetite and oxides of manganese to manganous oxide. The reduced ore discharged from the reducing furnace through a water seal will be stored in a bin. A bucket elevator and a vibrating feeder will feed the reduced ore to a double roll crusher to grind the material to about 10 mesh. The crushed material will subsequently pass through a wet magnetic separa-

tor to yield a magnetic product and a non-magnetic manganese concentrate. If finer grinding is required, the roll crushed material will be ground in a rod mill in close circuit with a classifier. The fine manganese concentrate will be dewatered in a thickener and the thickened pulp filtered in a filter. The filter cake will be dried in a rotary drier and stored. The concentrate will be either sintered or briquetted. This flowsheet will be suitable for ferruginous manganese ores low in silica and alumina. But the layout of the plant is so designed that other types of ores can also be investigated after suitable adjustment of necessary equipment.

## 58.0 Hot-dip Aluminizing of Steel Wire and Sheet

The importance of aluminizing in preference to galvanizing is now well established apart from the consideration that in India resources of zinc are considerably less abundant than aluminium and also the country's entire requirement of zinc is at present imported. A research scheme was taken up to obtain a satisfactory coating of aluminium over ferrous material by hot-dip processes and the experimental work resulted in a satisfactory coating by these different processes differing basically in the uses of fluxes such as molten salt, aqueous and organic fluxes. A pilot plant has been set up with

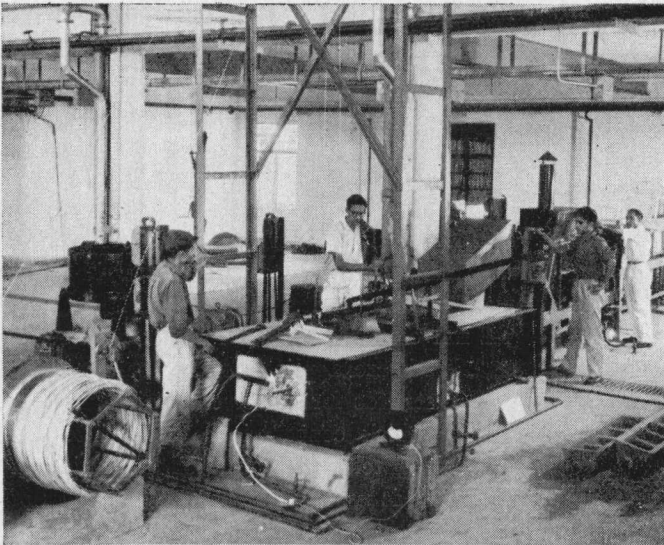


SCHEMATIC 5-STRAND M.S. WIRE HOT-DIP ALUMINIZING PILOT PLANT LAYOUT

a view to determine the commercial feasibility and economic aspects of the patented processes developed at the National Metallurgical Laboratory.

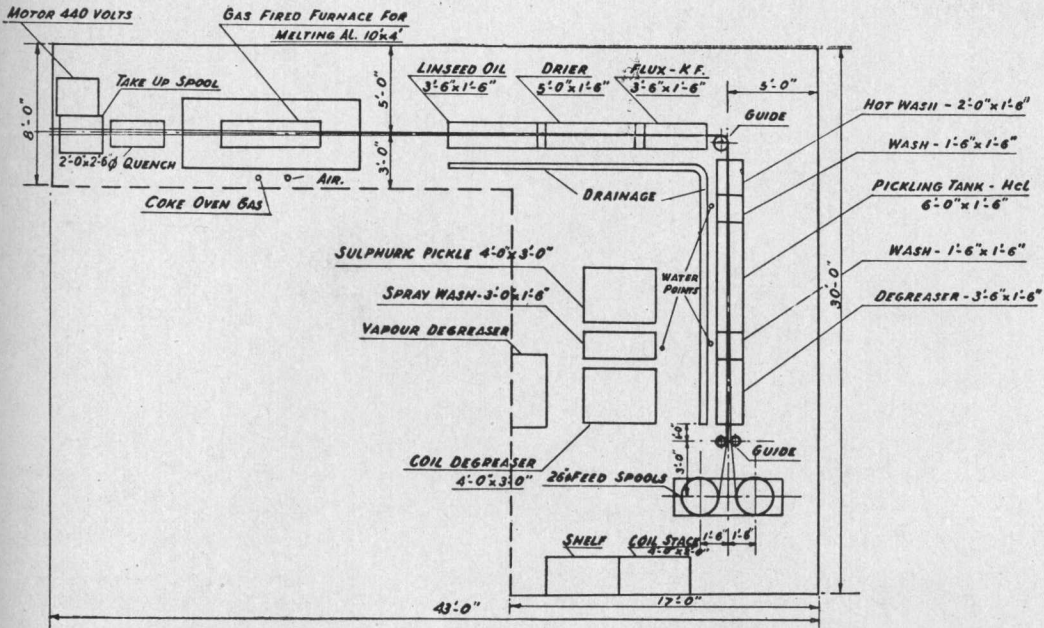
The set-up installed for aluminizing steel wire is also capable, with modifications, of strip aluminizing and is about  $\frac{1}{6}$  the size of the commercial wire-galvanizing plant operating at Jamshedpur. The pilot plant has five wire strands. The time of contact in constituent units such as pickling, washing, drying, etc., can be adjusted. The degreasing unit set-up will permit a longer time of contact without increasing the flow span required for the bath length. An alloy cast iron pot, resistant to high temperature and not so reactive towards aluminium, has been used for holding molten aluminium. The sinker required to keep the wire in the molten aluminium bath is also a casting of the same composition as the pot.

The plant uses annealed M.S. wire as the basis material to be coated. The coils are first degreased in I.C.I. degreaser No. 1, washed in running water and placed on feed



PILOT PLANT FOR HOT-DIP ALUMINIZING OF STEEL WIRE INSTALLED AT THE NATIONAL METALLURGICAL LABORATORY

reels. Wire from the feed roll enters an alkathene-lined pickling tank wherein wire is pickled in 1 : 1 HCl at room temperature. It is then washed free of smut and introduced



M.S. WIRE ALUMINIZING PILOT PLANT LAYOUT

in a fluxing tank. The flux employed is a 35 per cent solution of potassium fluoride at a temperature of 70°-80°C. The fluxed wire is passed through a drier and preheater heated by means of the flue gases from the gas-fired aluminium melting furnace. Fluxed wire enters the molten aluminium bath at 750°C. and is withdrawn vertically. The emerging wire is wiped with asbestos to avoid dross from sticking to it. Wiped wire has a thin 1-2 mil coating suitable for corrosion resistant hardware applications and core wire of ACSR strands for power transmission. For getting thick concentric coatings intended for use in telephone and telegraph lines a sizing die is used to control the thickness of coating on the emerging wire. The vertically withdrawn wire travels up 10-15 ft. in air before touching any pulley. After air cooling the wire is coiled over take-up reels.

The present set-up draws wire at 20 ft. per minute through the aluminium bath. Two wires are run simultaneously at present but the plant can be expanded to a five-strand line.

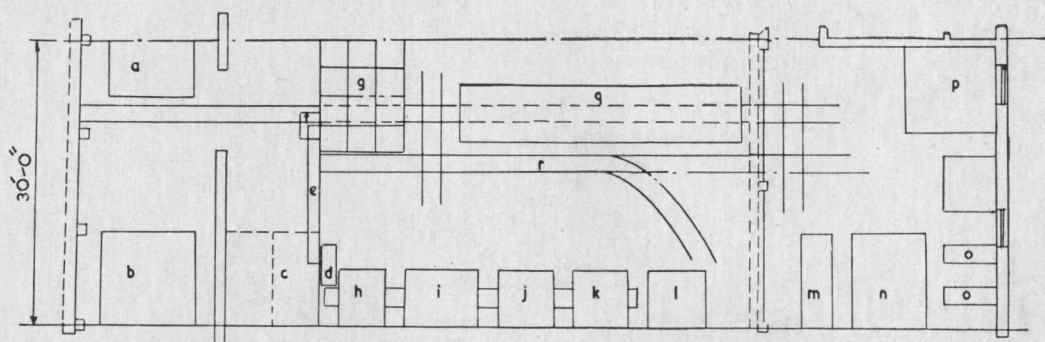
## 59.0 Pilot Plant for Refractories

Based on research carried out in the laboratory, a number of processes were developed on improved manufacturing techniques and

products of refractories which are not being made in the country so far. As pilot plant trials are very essential before such new products and techniques are exploited commercially, a pilot plant for the production of the following types of refractories is under installation:

1. Dense mullite and hot face insulation refractories from bladed kyanite
2. Zircon and sillimanite refractories from Travancore beach sands
3. Magnesite refractories from Almorah magnesite
4. Forsterite refractories from magnesium-silicate rocks
5. Chemically bonded and metal clad mixed basic refractories
6. Stabilized dolomite refractories
7. Chrome-magnesite refractories from low-grade chromites
8. Carbon refractories from petroleum coke and low ash coal
9. Dense carbon aggregates and Soderberg paste from indigenous raw materials

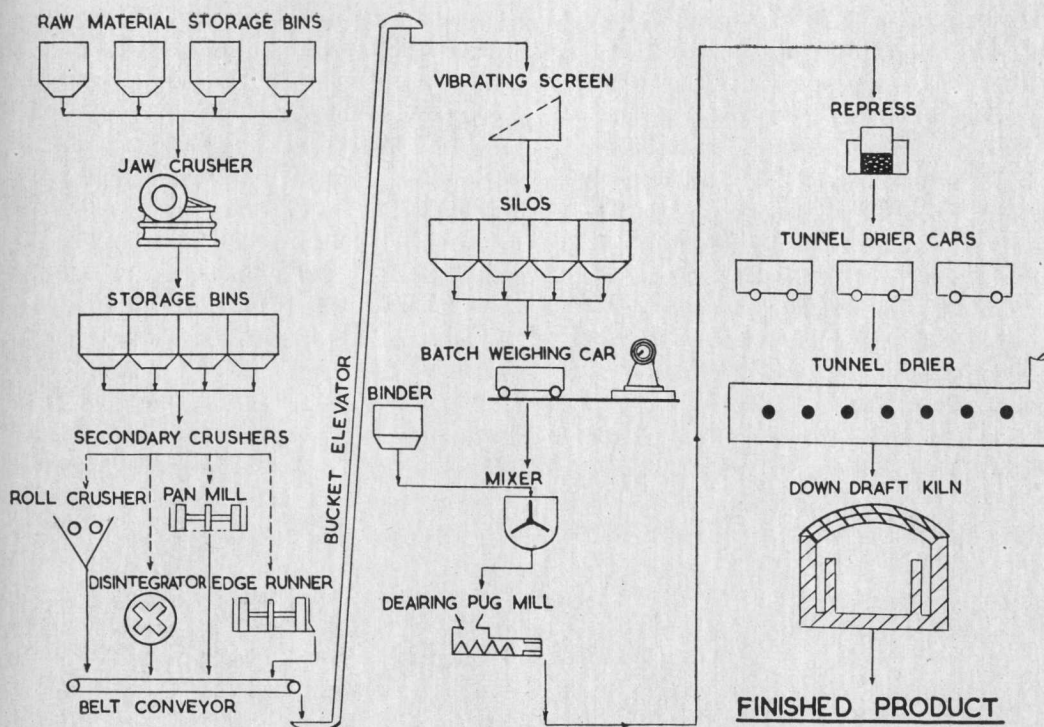
This pilot plant has been so designed that the various stages inherent in refractory manufacture can be repeated on a semi-industrial scale either unit-wise or in a continuous 'raw material to finished product' flow line. The unit is designed to handle a little over one ton of raw material per day of



a, Jaw Crusher. b, Down Draft Kiln. c, Bins. d, Elevator. e, Belt. f, Vibrating Screen. g, Silos. h, Roll Crusher. i, Disintegrator. j, Pan Mill. k, Edge Runner. l, Mixer. m, De-airing Plug Mill. n, Repress. o, Tables (Hand Moulding). p, Toggle Press. q, Tunnel Drier. r, Rail Track.

LAYOUT OF REFRACTORIES PILOT PLANT





FLWSHEET FOR REFRACTORIES PILOT PLANT

eight hours and comprises of a crushing and grinding section, a mixing and forming section and a drying and firing section. A casting section is also being included. Almost all the equipment are expected to be instrumented adequately so that collection of process data is rendered easy and practicable.

The layout of the pilot plant bay was finalized and a number of ancillary equipment necessary for running the pilot plant were thought out and their full specifications were drawn up. Most of these equipment have either been ordered for or are in the process of being ordered.

Similarly, specifications for specialized equipment like kilns and driers have also been drawn up. Some of the crushing and grinding equipment, which have been received, were installed in the pilot plant bay. Adaptation of the 500-ton hydraulic press for brick making is also progressing.

## 60.0 Pilot Plant Study on Recovery of Vanadium Pentoxide from Vanadiferous Magnetite Ores

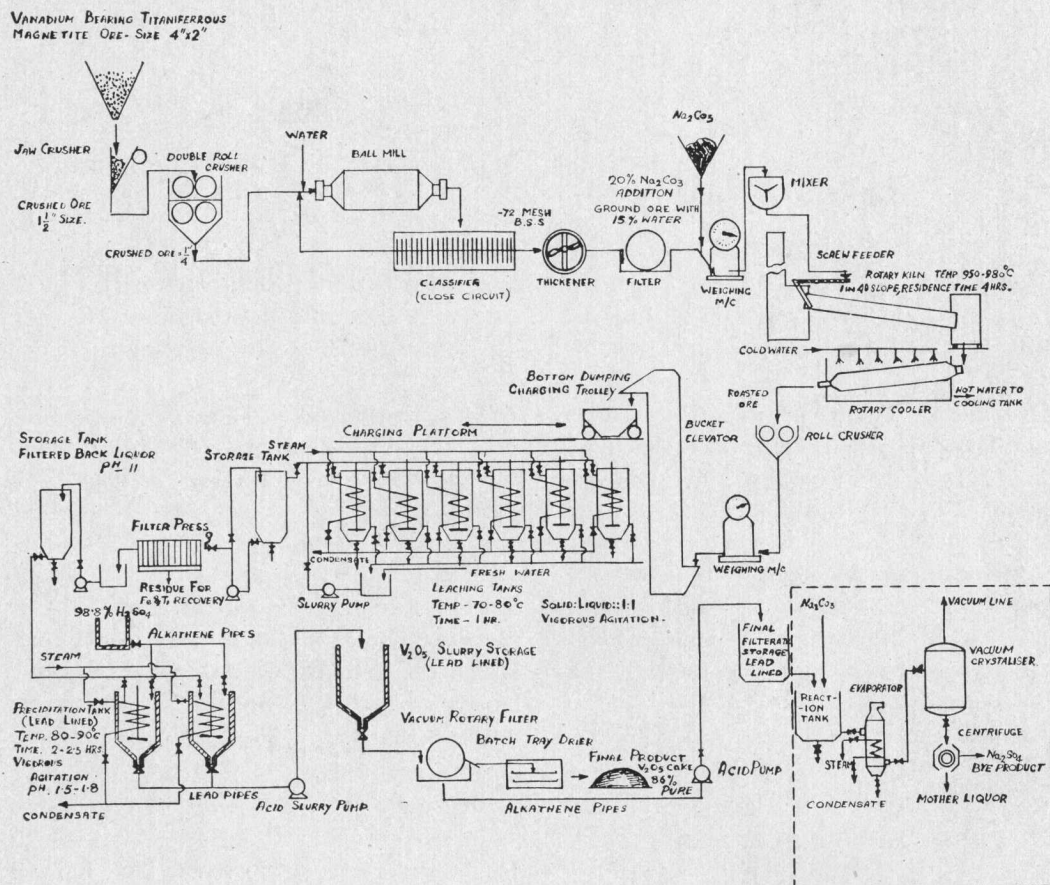
Vanadium containing titaniferous magnetites occur in Bihar and Orissa and their reserves have been estimated at about 22 million tons. The ore contains an average of Fe, 55-65;  $\text{TiO}_2$ , 8-12; and V, 0.8-1.8 per cent. These deposits owe their importance primarily to vanadium content. A systematic study for recovery of vanadium pentoxide was undertaken. Based on the laboratory scale experiments, a pilot plant for the treatment of one ton of ore per day is being set up.

The process consists in roasting of vanadium ores crushed to 72 mesh B.S.S. sieve with 20 per cent soda ash at  $950^\circ\text{C}$ . in a rotary kiln thereby converting the vanadium into sodium vanadate. The roasting of

vanadium ores will be carried out in a rotary kiln, lined with sillimanite bricks, 30 ft. long and 3 ft. in diameter. The kiln has been designed keeping in view that it should not only be suitable for roasting vanadium ores, but should also be capable of reduction roast studies for other ores. The roasted ore is crushed and leached in a series of six steam-heated tanks. The leaching tanks,  $5\frac{1}{2}$  ft. high and  $2\frac{1}{2}$  ft. in diameter, are provided with conical bottom and are made of mild steel. The tanks are also provided with water inlet, slurry outlet and flush-out openings. The slurry is agitated with mechanical agitators operating at various speeds. Leaching is carried out in five steps on the counter-current principle and the enriched solution

is pumped from one tank to another. The residue left in the tank in turn is re-digested with stronger solution. The enriched liquor containing 15-20 gm. of vanadium per litre as sodium vanadate is pumped into the lead-lined precipitation tanks and vanadium pentoxide is precipitated at 90°-95°C. by adjusting the *pH* to 1.5-2 with the sulphuric acid. The pentoxide is filtered on a filter press and dried and the depleted liquor is sent to crystallizers for the recovery of Glauber's salt.

The other aspects of the study on pilot plant will be the recovery and utilization of the byproducts, e.g. titania-rich slag and solution containing sodium sulphate and unrecovered vanadium pentoxide.



FLOW-DIAGRAM OF THE PROPOSED  $V_2O_5$  PLANT

## 61.0 Pilot Plant Study on Thermal Beneficiation of Low-grade Manganese Ore

The specification of manganese ores for metallurgical purpose requires Mn:Fe ratio should be as high as possible and preferably not below 7:1. The phosphorus content should also not exceed 0.1-0.13 per cent and the silica and alumina contents of the ore should not be more than 8 and 10 per cent respectively. The process of thermal beneficiation employed for the treatment of low-grade manganese ore is undertaken in a two-stage process, some work on which has already been carried out in the laboratory. The process consists in treatment of low-grade manganese ore through suitable thermal techniques aiming at the production of an enriched manganese slag and iron as a byproduct in the first stage, followed by the smelting of enriched manganese slag preferably in an electric furnace of soderberg type. On commercial scale, the possibility of a combination of acid Krupp-Renn rotary kiln during the first stage to produce an enriched slag followed by the use of basic Stuzelberg rotary furnace to yield a manganese-rich ferro-alloy in a second stage will be considered. Alternatively, electric smelting of enriched manganese slag to produce ferro-alloy may also be considered. The method is based on studies of free-energy values of the oxides of manganese, iron and phosphorus and thermo-dynamic factors involved in their reduction under optimum conditions of temperature and basicity. Pilot plant scale study of the process is expected to yield very valuable data on the various aspects of the process and a pilot plant is being installed in this laboratory. The furnace will have an initial capacity of 100 lb.

## 61.1 Pilot Plant Study on Thermal Beneficiation of Low-grade Chrome Ore

Suitability of chromite in the production of standard grade ferro-chrome limits the Cr:Fe ratio to 3:1 with a minimum of 48 per cent  $\text{Cr}_2\text{O}_3$ , MgO and  $\text{Al}_2\text{O}_3$  should not exceed 5 per cent. The Indian chrome ores are characterized by their high iron contents occurring in chemical combination with chromite. Conventional methods of ore-dressing or roast reduction followed by magnetic separation have not been found to improve the Cr:Fe ratio, even though a substantial increase in the  $\text{Cr}_2\text{O}_3$  content of the ore may be obtained. The thermal beneficiation process investigated in this laboratory consists in selective reduction of iron in the chrome ore with solid or gaseous reducing agents and its subsequent removal by acid leaching. A pilot plant based on this investigation is being designed and installed.

The chrome ore along with coke will be fed in a rotary kiln, 30 ft. long and 3 ft. in diameter. The maximum temperature at the firing zone will be about  $1300^\circ\text{C}$ . and the inclination of the kiln is so adjusted that a retention period of  $2-2\frac{1}{2}$  hr. is obtained in the high temperature zone. The reduced ore will be fed into a cooler and cooled in neutral atmosphere. The cooled reduced mass will be crushed to -10 mesh and fed into a lead-lined steam heated tank,  $2\frac{1}{2}$  ft. in diameter and  $5\frac{1}{2}$  ft. in height, for leaching. The leached mass will be taken into a thickener and a residue enriched in Cr:Fe ratio will be obtained. The liquor containing ferrous sulphate and dissolved salts will be used for the recovery of ferrous sulphate and subsequently for production of ferric oxide by roasting.



## LIAISON AND INFORMATION SERVICES

The liaison, information and publication work have considerably increased due to the heavy quantum of work relating to the publication of *NML Technical Journal*, proceedings of the Symposia, commercial exploitation of patents, liaison with industries and scientific institutions, collection of statistical data, technical information and dissemination of scientific research results to the industries, release of press notes, compilation of survey reports on subjects of topical value, etc.

Industries, State Governments and other organizations continued to call heavily with a large number of requests for technical assistance by way of technical information, process details, assessment of indigenous raw materials, flowsheets, beneficiation techniques for low-grade ores, heat treatment schedules, survey reports, examination of failures of metals and alloys in service and corrosion of metals and alloys under specific environments. The technical aid to industries often requires exhaustive examination of technical information available from published sources and at times also requires to be supplemented with *ad hoc* investigations from the respective research divisions of the laboratory. In these activities, Liaison and Information Division played an active role. Periodical statistical survey on production, import and export of various mineral and metal products was also made from time to time.

In the field of dissemination of research to industries, non-technical notes of the processes developed in the laboratory were prepared and widely circulated for their commercial exploitation. Practical demonstrations were held to appraise the industries, the actual know-how of the process. Popular

brochures were brought out periodically highlighting the achievements of the laboratory and press releases were made for important announcements regarding the developments from time to time.

The *NML Technical Journal*, which was started a year ago as a quarterly technical publication, has now stepped into the second year of renewed technical features providing a vital link between the industry and scientific organization. The response to the journal from advanced metallurgical and scientific organizations in the world has been not only inspiring but also encouraging.

The Symposium on "Pilot Plants in Metallurgical Research and Development" was organized during the year under review which was attended by the leading scientists and technologists from all over the world. The abstract of all the papers presented during the Symposium together with a few papers in full were published and circulated to all the delegates much in advance of the Symposium to stimulate discussions during the technical sessions. A short proceedings of the deliberations of the Symposium was prepared immediately after the Symposium and was circulated to all the technical journals in India and abroad. Many of the technical journals published a full account of the proceedings. During the year under review, the proceedings of the Symposium on "Iron and Steel Industry in India", which was held under the auspices of the National Metallurgical Laboratory in February 1959, was brought out within a record time of four months. The publication, containing 45 authoritative technical papers brought out with a full account of the discussions during the Symposium, was widely appreciated by all the delegates and was also reviewed in

many of the technical journals at home and abroad.

A monograph on beneficiation of low-grade manganese ores in India based on the exhaustive investigation work carried out by the laboratory on the low-grade manganese ores, on all-India basis, was also brought out which was reviewed most favourably by the technical journals in India and abroad. The monograph on foundry moulding sands and bonding clays in India containing a comprehensive account of the various foundry sands and bonding materials available in the country is under publication.

Weekly colloquium was arranged in which the research workers and other staff members

of the laboratory were given an opportunity to give an account of the work done by them for the benefit of others. The brief account of the multifarious work of the division during the period under review is furnished below.

## TECHNICAL AID TO INDUSTRIES

Technical advice was rendered to various industrial organizations and Government bodies. 127 technical enquiries were attended and 12 short-term investigations and specification tests on behalf of the industries were conducted during the period under review. Details are furnished below:

### Technical Enquiries

<i>Sl. No.</i>	<i>Name of the Party</i>	<i>Nature of Assistance Rendered</i>
1.	M/s Hercules Emery Works, Belgaum	Manufacture of thermostatic expansion valves
2.	M/s M. R. Salivan & Sons, New Delhi	Composition and heat treatment of steel spring wires
3.	M/s Laxmi Jewellers, Srinagar	Manufacturing process of rolled gold and the imitation gold alloy
4.	M/s E. E. A. Daniels, New Delhi	Details of reference books on stainless steel
5.	M/s Hindustan Metals Works, Hathras, U.P.	Refining of non-ferrous metal scraps
6.	Mr. Davindera Kumar Dhingra, Ludhiana	Names and addresses of suppliers of electric furnaces for hardening high speed steel
7.	M/s Bishamber Dass & Sons Ltd., New Delhi	Information regarding lacquering
8.	M/s Jyoti Ltd., Baroda	Information on manufacturing of 'Elmet' electrical contact
9.	M/s Madhav Electrical Industries, Bombay	Information regarding bimetal strip
10.	Mr. Tribhavan Nath Sain, Hoshiarpur	Details of reference books on stainless steels
11.	M/s Indophilips Industries Ltd., Calcutta	Details regarding magnet alloy
12.	M/s Kusum Engg. Co. Ltd., Calcutta	Chemical composition and heat treatment of steel for anvils
13.	M/s Daya Ram Shikkarchand, Delhi	Process of melting stainless steel
14.	M/s Automobile Products Co., Agra	Details of books on wire and wire products
15.	M/s Bicky Roberts (India) Corpn., Jullunder City	Composition of the platinum alloy used for valve faces and seats

<i>Sl. No.</i>	<i>Name of the Party</i>	<i>Nature of Assistance Rendered</i>
16.	Villait Industries, Calcutta	Details of anodizing process
17.	Electrodes ( India ) P. Ltd., Calcutta	Processes of electroplating and chemical polishing of metal sheets
18.	M/s A. B. Brothers, Ahmedabad	Addresses of furnace suppliers
19.	The Malleable Iron & Steel Castings, Bombay	Availability of petroleum coke
20.	Shri T. S. Nagarathanam, Trichi	Composition and heat treatment of permanent magnet alloy steel
21.	Mr. M. S. Subramanyam, Chief ( Industry ), Planning Commission, Govt. of India, New Delhi	Details of electrochemical refining of lead
22.	Shri Talagadadevi Prasad Rao, The Andhra Scientific Co. Ltd., Masulipatam	Some details about gun metal composition and casting
23.	The Mahalaxmi Industries, Bhavnagar	Processes of blueing of steel ( nails ) and name of suppliers of furnaces
24.	The Director, Central Tobacco Research Institute, Rajahmundry	Electronic devices of non-destructive tests and its literature reference
25.	The General Manager, P. & T. Workshops, Calcutta	Procedure of measurement of thickness and uniformity of zinc coating and testing, and procedure of testing electro-deposited zinc and copper castings
26.	M/s Siacco (P) Ltd., Bangalore	Composition of magnet alloys for cycle dynamos
27.	Dr. M. R. Mandlekar, Director of Industries, Bombay	Suitability of some type of iron ores for the production of ferro-alloys
28.	M/s National Pipes & Tubes, Calcutta	Addresses of suppliers of nitrided steel
29.	M/s Ganga Saran & Sons (P) Ltd., Calcutta	The manufacturing process of iron and copper alloy and the address of its suppliers
30.	M/s M. V. Raste, Poona	Outline of the process of manufacture of ferrous sulphide
31.	M/s Royal Optical Industries, Calcutta	Details of the process of manufacture of rolled gold
32.	The Asst. Works Manager, Indian Ordnance Factories, Ambarnath	Addresses of suppliers of bakelite powder
33.	M/s Hindustan Metal Works, Hathras	Reference books on metallurgical analysis
34.	Dr. K. M. Chakravarty, Ranchi	Literature reference on carbon steel and 18/8 steel
35.	Shri S. P. Sinha, Calcutta	Utilization of waste material like aluminium foils and aluminium scrap
36.	The Indian Standards Institution, New Delhi	Specification of mineral sample for the determination of the hardness of minerals
37.	The New Friend & Co., Delhi	Compositions of brass used for the manufacture of clocks



<i>Sl. No.</i>	<i>Name of the Party</i>	<i>Nature of Assistance Rendered</i>
38.	The Chhotanagpur Engg. Works, Ranchi	Addresses of suppliers of laboratory equipment
39.	The Poona Auto Works, Poona	Names and details of books on chrome plating
40.	M/s Metal Moulding & Pressing Works, Bombay	Some precautions to be taken in melting stainless steel scrap
41.	M/s Mohindra Engg. Corpn., Jullundur City	Type of kyanite suitable for manufacture of fire cement
42.	M/s Indo Phillips Industries (P) Ltd., Calcutta	Manufacturing process of magnet alloys
43.	M/s Saxby & Farmer ( India ) (P) Ltd., Calcutta	Names and addresses of suppliers of carbon standards, potassium bisulphate and sulphur dioxide
44.	M/s Indian Graphite Industries, Vijayawada	Utility of steatite
45.	M/s Precision Metal Industries, Bombay	Names and details of books on remelting and refining of M.S. scraps and the addresses of the consulting engineering firms
46.	Mr. R. Sharma, Tumsar Road	Information about the manufacturing process for high carbon ferro-chrome
47.	Mr. Narayanswamy, Small Industries Service Institute, New Delhi	Information about malleable castings, design of annealing furnace, mixture of iron to be used to melt in crucible for B.B. Shell and Auto parts and design of containers
48.	M/s Refrigeration & Appliances Co. (P) Ltd., Bombay	Different compositions of steels suitable for scaling resistance at 800°C.
49.	Major M. Dutta, Calcutta	Process of hard chromium plating
50.	The Asst. Director, Small Industries Service Institute, New Delhi	Compositions of the fluxes and wires for arc welding electrodes
51.	M/s Poona Auto Works, Poona	Manufacturing process of magnet alloys
52.	M/s Rashtriya Metal Industries Ltd., Bombay	Casting defects and their remedies
53.	Shri R. P. Jadhav, Jadhav Refinery, Kolhapur	Names of books on silver plating
54.	The Director, Reliance Firebricks & Pottery Co. Ltd., Calcutta	Information on the manufacture of welding fluxes from Indian kyanite
55.	The Joint Director of Industries, Bombay	Compositions of different stainless steels suitable for making moulds for plastic mouldings
56.	Metal Engineering Works, Jullundur City	Names of books on case hardening
57.	M/s Baroda Rolling Works, Bombay	Method of removing lead impurity while melting of brass scrap
58.	Mr. M. R. V. Rajgarhia, Calcutta	Definition of alloy steel

<i>Sl. No.</i>	<i>Name of the Party</i>	<i>Nature of Assistance Rendered</i>
59.	Shree Metal Industries, Moradabad, U.P.	Composition of German silver and literature references on it
60.	M/s Ramchandra Shankar Lal, Mirzapur, U.P.	do
61.	The Assistant Secretary, The Institution of Engineers, Calcutta	Composition of steels used for automobile valve
62.	M/s National Instruments Ltd., Jadavpur, Calcutta	Hardness and chemical composition of silver strips
63.	M/s Swadesh Engg. Co., New Delhi	Composition of heat-resistant alloys
64.	Shri S. S. A. Nadar, Tuticorin	Details of the processes of blueing of mild steel
65.	M/s Hindustan Aircraft ( Private ) Ltd., Bangalore	Name of supplier of crucibles
66.	M/s Fasteners & Wire Products, Lucknow	Information regarding: (a) Annealing of rivets (b) Suitable die steel for code punch
67.	M/s Khandalwal Bros., Ltd., New Delhi	Statistical data about spring steel tubes and pipes
68.	M/s Electro Metallurgical Works (P) Ltd., Bombay	Information on thermite mixture for welding steel
69.	M/s Iron, Steel & Hardware Merchants' Chambers of India, 'Steel Chamber', Bombay	Uses of pig iron
70.	Shri K. N. Poddar, Nagpur	Utility of Dolomite and fireclay, and names of parties who may be interested in party's materials
71.	Shri S. Arumugaswamy Nadar, Tuticorin	Names and addresses of manufacturers of steel of Siemens Martin quality
72.	Dr. N. S. Pandya, Physics Dept., S.J. Science Institute, Baroda	Names of suppliers of bismuth metal
73.	The Regional Chamber of Commerce, Santhapet, Nellore ( India )	Addresses of the engineering firms and suppliers of aluminium metal
74.	M/s Navin Stores, Ahmedabad	Information about cigarette lighter flints and literature references for the manufacture of misch metal
75.	M/s P. V. Kotwala & Co., Bombay	do
76.	Mr. D. Rohatgi, Bombay	Information about silica sands and names of suppliers of refractory bricks
77.	M/s Hind Harmonical Industries, Rajkot	Literature references on anodizing of aluminium
78.	M/s Bhagavati Prasad Baijnath Prasad, Mirzapur, U.P.	Precautions to be taken while melting during preparation of standard high carbon ferro-manganese
79.	The Works Manager, Integrated Coach Factory, Madras	Names of suppliers of alloy steel castings

<i>Sl. No.</i>	<i>Name of the Party</i>	<i>Nature of Assistance Rendered</i>
80.	M/s United Metal Industries, Calcutta	Literature references on anodizing of aluminium
81.	Deputy Director ( Chemicals ), Indian Standards Institution, New Delhi	Names of suppliers of test strips of required standard
82.	The Supdt., Indian Ordnance Factories, Ambarnath	Names of suppliers of Seger cones
83.	The Director of Mines & Geology, Govt. of Bihar, Patna	Specifications of limestone fit for use in the iron and steel industry
84.	M/s Bharat Minerals Industries (P) Ltd., Nagpur	Price of manganese metal in U.K. and U.S.A.
85.	M/s Unipupex (P) Ltd., Bombay	Details of the manufacturing process of malleable castings
86.	M/s Kusum Engg. Co. Ltd., Calcutta	Some details about heat treatment of cast steel anvils
87.	Shri N. Raman, Model Foundry, Madras	Types of melting furnaces used for malleable and steel foundry
88.	M/s Dusson Industries, Poona	Composition and heat treatment of die steel
89.	The National Iron Foundry, Agra	The composition of some specific type of cast iron
90.	The Secretary, Institute of Indian Foundrymen, Calcutta	Composition and availability of foundry coke, limestone and sand
91.	M/s Sanders & Miners, Dehra Dun, U.P.	Names of suppliers of jaw crushers
92.	M/s Rajasthan Industries, Falna	Names of suppliers of MnO <sub>2</sub> and zinc sheet
93.	M/s India Hardware Industries Ltd., Bombay	Qualities of pig iron suitable for casting power press parts
94.	The Director, Central Leather Research Institute, Madras	Composition of Martin's steel and its availability
95.	M/s Chingan Ram Musai Ram, Mirzapur, U.P.	Composition of Tripoli metal polish
96.	M/s Kirloskar Bros. Ltd., Kirloskarvadi, South Satara	Types of binding materials used for foundry purpose
97.	M/s Lalbhai Patel & Co., Bombay	Names of suppliers of killed steel
98.	Dr. G. B. Deedhar, Allahabad University, Allahabad	Names of suppliers of spectroscopically pure metals and alloy
99.	Shri S. Dutta, Calcutta	Composition of the heat-resistant cast iron
100.	Shri N. Raman, Asst. Engineer, Model Foundry, Guindy, Madras	Types of melting furnaces used for malleable and steel foundries
101.	The Director, Defence Metallurgical Laboratory, Ishapur, West Bengal	Some information on grit chilled iron
102.	Shri K. N. Murthy, Madras	Details of the manufacturing process of permanent magnets



<i>Sl. No.</i>	<i>Name of the Party</i>	<i>Nature of Assistance Rendered</i>
103.	M/s Fairplay Chemical Industries, Baroda	Manufacturing details for electrolytic iron powder
104.	M/s Saru Smelting & Refining Corpn. (P) Ltd., Meerut	Detail of continuous casting process
105.	Mr. K. K. Nathani, Textile Machinery Corpn., Calcutta	(a) Chemical composition of SAE-3140 steel and its heat treatment (b) Chemical composition of ASTM-A/148-58 steel and its heat treatment
106.	Mr. M. L. Sethi, Director of Mines & Geology, Udaipur	Some details about setting up of ferro-alloy plant like ferro-tungsten, etc.
107.	Dr. Bunshah, Bombay	Statistics about the expected demand of malleable casting from 1961 to 1976
108.	M/s Phool Chand, Delhi	Method of assaying of silver samples
109.	M/s Khandelwal Ferro-Alloys Ltd., Kamptee	Addresses of suppliers of metallurgical illustrated charts
110.	M/s Andhra Scientific Co. Ltd., Masulipatam	Names of books on annealing of electro-deposited material
111.	Shri Y. N. Trivedi, Patna	Composition and other specifications of steel used for chaff cutter.
112.	Dr. Prem Swarup, Allahabad	Address of the suppliers of Armco and Low Moore iron
113.	M/s Sudarshan Industries, Gurgaon	Address of suppliers of the shaft straighteners
114.	M/s Ramnarain Harcharanlal, Farrukhabad	An outline of the technique of melting of copper and brass
115.	M/s Barakar Engg. & Foundry Works Ltd., Dhanbad	Composition of the machinable iron castings
116.	M/s Bird & Co. (P) Ltd., Calcutta	Some information regarding fatigue testing of spindles
117.	Industrial Liaison Officer, C.S.I.R., Jaipur	Material required and the process of manufacture of gas cylinders
118.	M/s Shiv Scientific & Chemicals, Agra	Names and addresses of the consulting engineers
119.	Shri R. M. Ramaswamy, Udamalpet	Chemical composition and heat treatment of leaf spring used for automobile vehicles
120.	Mr. Niharendu Mallick, Howrah	Ditinning process and the reference of books on tin
121.	M/s Mahandi Trading & Manufacturing Co., Cuttack	Some information about the manufacture of aluminium powder
122.	The Wesman Engg. Co., Calcutta	The procedure of glazing of reverberatory furnace lining
123.	Shri S. Dutt, Calcutta	Composition of heat-resistant cast iron
124.	M/s New Horizon, Pondicherry	An outline of the process of manufacture of electrical resistance wire

<i>Sl. No.</i>	<i>Name of the Party</i>	<i>Nature of Assistance Rendered</i>
125.	M/s Modern Foundry & Machine Works & Co., Ahmednagar	The origin and sources of bentonite
126.	The Atul Products Ltd., Bombay	Sources of bauxite in Bombay State
127.	Industrial Chemical Corpn., Delhi	Composition of salt baths used for heat treatment of high speed steel

### Short-term Investigations and Specification Tests

1.	M/s Premier Automobile Ltd., Bombay	Investigation on samples of malleable cast iron
2.	The Director, Designs, Bhakra Dam Designs Directorate, New Delhi	Investigation on the failure of central training wall of the Bhakra Dam
3.	M/s R. M. H. Corporation, Tiruchirappalli 2	Process of tempering of M.S. sheet of screen
4.	Shri Ram Institute for Industrial Research, New Delhi	Analysis of the fabric samples
5.	The Solem Hardware Mart, Structural Engineers, Bombay 3	Testing of hot-dipped galvanized M.S. strips according to B.S.S. 729
6.	The Plant Manager, Durgapur Coke Oven Plant, Durgapur, Burdwan	Analysis of the teeth of the crusher
7.	M/s Standard Batteries Ltd., Bombay	Analysis of lead dross sample
8.	M/s Parshuram Pottery Works Co. Ltd., Bombay	P.C.E. test of fireclay samples
9.	Shri Ram Institute of Industrial Research, Delhi	Analytical report of metal on fabric
10.	The Regional Inspector of Mines, Sitarampur	Metallurgical examination of capple.
11.	The National Engineering Ltd., Jaipur	Inclusion count of steel samples
12.	M/s Arim Metal Industries (P) Ltd., Calcutta	Determination of oxygen content of nickel-castings

### PUBLICATIONS

Nearly three hundred and fifty papers have been published up to date in Indian and foreign journals. The details of the publications during the year under review are furnished in Appendix I.

### PATENTS

During the year under review, non-technical notes on the following processes developed

in this laboratory were prepared and circulated:

1. An improved method for the production of titanium tetrachloride from ilmenite — Patent No. 58244
2. A process for the production of chemically bonded metal-clad or unclad basic refractories — Patent No. 65610
3. Refractory compositions containing non-refractory chrome ore and refractory products made therefrom—Patent No. 68174
4. Liquid gold ( non-patented method )

A large number of industries have expressed keen desire to utilize some of the above processes and offers for the commercial exploitation of the processes have also been received which are under consideration.

The following patents have been filed during the period under review:

1. A method for manufacture of porous bronze bearings — Patent No. 67871, filed on 1-6-1959
2. Compositions and method of making welding flux — Patent No. 68171, filed on 26-6-1959
3. Refractory compositions containing non-refractory chrome ore and refractory products made therefrom — Patent No. 68174, filed on 26-6-1959
4. Improvements in or relating to the manufacture of bricks or blocks out of ceramic mixes — Patent No. 68401, filed on 15-7-1959.

Patents for the following processes have been accepted during the year:

1. Refractory compositions comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation — Patent No. 62352, accepted on 8-8-1959
2. A process to produce dense carbon aggregates from carbonaceous materials of varied volatile contents — Patent No. 62938, accepted on 15-7-1959
3. A process for recovering zirconium dioxide from zircon — Patent No. 63904, accepted on 16-9-1959
4. An improved method for the production of chromium-manganese alloys by alumino-thermic reaction — Patent No. 65231, accepted on 6-8-1959
5. A process for electrolytic production of iron-chromium alloys from chromite ore — Patent No. 65556, accepted on 6-1-1960

The following patents were sealed during the year:

1. New stainless steel and methods of preparing them — Patent No. 61978

(cognated with 61979 and 61980), sealed on 9-9-1959

2. A process for making completely stabilized dolomite refractories — Patent No. 61981, sealed on 8-7-1959

The following patents have been released to industries on royalty and premia basis:

1. Refractory compositions comprising graphite and silicon carbide — Patent No. 58869
2. Refractory composition comprising graphite and alumino-silicate materials and glazes to render such compositions resistant to oxidation — Patent No. 62352
3. Improvements in, or relating to, mullite refractories from kyanite — Patent No. 58553

## Patent Service

A survey of published patent literature on metallurgical and related topics has recently been initiated with a view to keep the research workers abreast of the latest processes, techniques, apparatus and materials developed and patented in metallurgical field. Such survey will be of much assistance in the commercial applicability of laboratory's inventions and will also help in pursuing research work in fruitful directions.

The work on patent literature survey consists in

1. Sorting out patent literature on metallurgical topics from about 650 current scientific periodicals and patent specifications received monthly at the National Metallurgical Laboratory Library
2. Summarization and division-wise classification of the patent literature collected
3. Distribution of classified summaries to concerned divisions and sections of the laboratory
4. Arranging procurement of complete specifications for selected number of



patents for detailed examination by concerned research workers

5. Discussion and evaluation of patents of interest for National Metallurgical Laboratory in its research and development projects

## PRACTICAL DEMONSTRATION

The second demonstration programme of the following patented processes released free of royalty was held from 31st July to 7th August 1959 at the National Metallurgical Laboratory:

1. Improvement in, or relating to, electroplating of metals on aluminium — Patent No. 51524
2. Chemical polishing of aluminium — Patent No. 47401
3. Improvement in, or relating to, brass plating from non-cyanide bath — Patent No. 45565
4. Improvement in, or relating to, metallization of non-conductors — Patent No. 45579

Inaugurating the demonstration programme in the presence of a large number of representatives from small-scale industries, Sir Jehangir Ghandy, Kt., C.I.E., Director-in-Charge, Tata Iron & Steel Co. Ltd., and Chairman of the Executive Council, National Metallurgical Laboratory, observed that while the primary task of the laboratories is to carry out research, industrial utilization of the results of research should also be an equally important aspect of their work, if the fruits of research are not to be kept in the cold storage. He was happy to find that the National Metallurgical Laboratory is attaching great importance in this matter and extending facilities for the growth and development of small-scale industries, which are receiving good response. He thanked Dr. B. R. Nijhawan and his colleagues who are working hard in the pursuit of various research projects and their utilization.

Addressing the representatives of the industries, Dr. B. R. Nijhawan, Director,

National Metallurgical Laboratory, said that lump-sum payment offers were received which could have brought to the research worker a fair monetary return. But in the large interest of the small-scale industries, no royalty was charged and free demonstration of the processes was arranged to provide necessary technical know-how. The National Metallurgical Laboratory will arrange for such periodical demonstration which will act as a bridge between the industries and the National Metallurgical Laboratory.

In the week-long programme, the representatives of industries watched with all eagerness the demonstration and expressed keen interest in understanding the technicalities of the operational details of the methods.

## SYMPOSIUM

A Symposium on "Pilot Plants in Metallurgical Research and Development" was organized by the National Metallurgical Laboratory at Jamshedpur from 15 to 18 February 1960 to focus attention on the role of pilot plants in metallurgical research and development and with a view to exchange technical know-how with the leading scientists and metallurgists from different parts of the world. The Symposium was

SIR J. J. GHANDY, KT., C.I.E., CHAIRMAN, EXECUTIVE COUNCIL OF THE NATIONAL METALLURGICAL LABORATORY AND DIRECTOR-IN-CHARGE, TATA IRON & STEEL CO. LTD., DELIVERING THE PRESIDENTIAL ADDRESS ON THE INAUGURATION DAY OF THE SYMPOSIUM ON "PILOT PLANTS IN METALLURGICAL RESEARCH AND DEVELOPMENT"



inaugurated by Prof. M. S. Thacker, Director-General, Scientific & Industrial Research. Sir J. J. Ghandy, Kt., C.I.E., Director-in-Charge, Tata Iron & Steel Co. Ltd., and Chairman of the Executive Council of the National Metallurgical Laboratory presided.

Sir Jehangir Ghandy, in his presidential address, stated that the results of research investigations in the laboratory have to be re-examined on a pilot-plant scale in order to determine their feasibility for implementation on a production basis. He commended the considerable stress laid by the National Metallurgical Laboratory in translating its laboratory-scale research results on to pilot-plant scale with a view to determine their applicability in Indian mineral and metal industries. He made a reference to the valuable work being done at the pilot low-shaft furnace for the production of pig iron from low-grade ore and non-metallurgical fuels at the National Metallurgical Laboratory. He extended, on behalf of the Executive Council of the National Metallurgical Laboratory, sincere thanks and deep appreciation to Dr. B. R. Nijhawan, Director, and his colleagues for the valuable research and development work being done by them at the National Metallurgical Laboratory and also for the adequate arrangements made for the Symposium.

Prof. M. S. Thacker, Director-General, Scientific & Industrial Research, in his inaugural address stated that the Council of Scientific & Industrial Research was laying considerable stress in implementing laboratory-scale research results in industry through the medium of pilot-plant scale investigations and trials. He stated that some of the pilot-plant scale research results emanating from the National Metallurgical Laboratory had already found translation and application in industry. He emphasized the necessity of response from the industry to accept duly established and scientifically sound processes emanating from the National Laboratories even though there may be a period of teething troubles associated

with such implementation anywhere. Prof. Thacker expressed great satisfaction and paid a warm tribute to Dr. Nijhawan and his staff for the excellent work they were doing towards the development of metal and mineral industries of the country.

Dr. B. R. Nijhawan, Director, National Metallurgical Laboratory, in welcoming the distinguished delegates from India and abroad, stated that in diverse fields of metallurgical industries, art and craftsmanship are either being replaced or supplemented by a systematic study of the basic scientific factors involved and their co-ordinated application initially on a laboratory bench and then on to pilot-plant scale. Dr. Nijhawan further stated that the National Metallurgical Laboratory had recognized the importance of initiating pilot-plant projects based on laboratory scale research results with a view to latter's implementation in industry. He stated that the new pilot-plant bay being opened during the Symposium will contain four important pilot plants such as the aluminizing of steel wire and sheets, beneficiation of low-grade Indian manganese, chrome and other ores by ore dressing and thermal beneficiation techniques, production of electrolytic manganese and manganese dioxide from low-grade Indian ores and production of indigenous refractories for use in steel and metallurgical industries.

The Symposium drew a large gathering of top-ranking scientists and technologists from all over the world, like Dr. James B. Austin, Administrative Vice-President, Research and Technology, United States Steel Corporation, Pittsburgh, U.S.A.; Mr. Henry G. Iverson, Chief, Branch of Ferrous Metals and Ferro Alloys, Bureau of Mines, U.S. Dept. of the Interior, Washington, U.S.A.; Mr. Abbott Renick, Minerals Attache, American Embassy in India; Mr. E. W. Voice, Assistant Director, The British Iron and Steel Research Association, U.K.; Dr. N. Booth, Managing Director, The British Oxygen Research and Development Ltd., U.K.; Mr. M. D. J. Brisby, Chief Engineer, Messrs W. S. Atkins & Partners,



DR. B. R. NIJHAWAN, DIRECTOR, NATIONAL METALLURGICAL LABORATORY, WELCOMING THE DELEGATES TO THE SYMPOSIUM ON "PILOT PLANTS IN METALLURGICAL RESEARCH & DEVELOPMENT" ON THE INAUGURATION DAY

London, U.K.; Dr. A. Grieve, Incharge, Research & Development, Huntington Heberlein & Co. Ltd., Stockport, U.K.; Dr. Ing.habil Hans Heinrich, Indien Gemeinschaft Krupp-Demag GmbH, Duisburg, W. Germany; Dr. Hans Rausch, Lurgi Gesellschaft fur Chemie und Huttenwesen mbH, Frankfurt/Main, W. Germany; Mr. Jacques Astier, Chef de Service Development, Institut de Recherches de la Siderurgie (IRSID), Station d'Essais a Maizières-Les-Metz, France; Mr. Albert Bureau, Honorary Director, Ministry of Industry (Paris) and Adviser to French Iron & Steel Federation, Government of France; Mr. D. H. Hart, Technical Asst. to Manager, The Broken Hill Associated Smelters Pty. Ltd., Port Pirie, South Australia; Prof. N. I. Shirokov, Unesco Expert and Prof. of Ferrous Metallurgy, Indian Inst. of Technology, Bombay; Prof. M. A. Maurakh, Unesco Expert and Prof. of Non-

ferrous Metallurgy, Indian Inst. of Technology, Bombay; Prof. G. Reginald Bashforth, Unesco Expert in Metallurgy and Professor of Ferrous Metallurgy, Banaras Hindu University; Mr. J. G. Goetzee, Process Development Engineer, Bataafse Internationale Petroleum Maatschappij N.V., The Hague, Holland; Mr. A. Honda of Yawata Iron & Steel Works, Yawata, Japan; etc.

Twenty-seven technical papers covering the various aspects of pilot plant work in metallurgical research and development were presented and discussed.

Synchronizing with the Symposium session, Prof. M. S. Thacker opened the new Pilot Plant Technological Building of the National Metallurgical Laboratory. He also gratefully acknowledged a gift worth over a million rupees of equipment received from the British Government under the Colombo Plan which were ceremoniously handed over to the



G. W. Brazendale, Trade Commissioner-in-Charge of the U.K. High Commissioner's Office in India.

Colloquia were held on the following subjects during the year.

<i>Subject</i>	<i>Speaker</i>
1. Kinetics of the pyrolysis of bituminous coal at low temperature	Mr. P. Prabhakaram
2. Problems related to the development of bi-metal	Mr. J. Bhattacharyya
3. Foundry sand and sand control	Mr. J. Mohan
4. Semi-pilot plant study on the beneficiation of ferruginous manganese ore from Siljora Kalimati Mines, Orissa, by magnetizing reduction process	Mr. B. L. Sengupta
5. Installation and commissioning of electrical power substations	Mr. H. Singh
6. Development of electrical resistance alloys for heating element	Mr. R. Choubey
7. Aluminium coating by immersion coating	Mr. A. N. Kapoor
8. Aluminium coating by electrophoresis	Mr. A. N. Kapoor
9. Production of low-carbon ferro-chrome	Mr. V. S. Sampath
10. Preparation of electrolytic iron-chromium alloys from chromite ore	Mr. P. B. Chakravarty
11. Fabrication of vacuum fusion apparatus and its working	Mr. N. G. Banerjee
12. Austenitic transformation and the Greninger-Troiano technique	Dr. E. G. Ramachandran
13. Recent trends in electroplating	Mr. D. S. Tandon
14. Continuous casting	Mr. B. V. Somayajulu
15. Zircon refractories from Travancore sands	Mr. M. C. Kundra
16. Talk about the deputation to U.K.—Sintering and pelletizing of ore	Mr. G. P. Mathur

## AWARDS, DEPUTATIONS AND NOMINATIONS

### AWARDS

Shri A. Ghosh, J.S.A., has been awarded "Captain N. N. Dutta Medal" by the Council of Chemists (India), for securing highest marks in aggregate at the Associateship examination held in November 1959.

### DEPUTATIONS

1. Dr. B. R. Nijhawan, Director, was deputed to France under the Indo-French Economic and Technical Co-operation Agreement. He also visited various research institutions, industries, etc., in Belgium, U.K. and Germany.

2. Shri P. I. A. Narayanan, Asst. Director, was deputed to France for training under the Indo-French Technical Co-operation Agreement.

3. Shri B. L. Sen, S.S.O., was deputed to West Germany for training in the operation and maintenance of low-shaft furnace pilot plant.

4. Shri G. P. Mathur, S.S.O., was deputed for training in the field of ore beneficiation, sintering and pelletizing of iron ores, under the Colombo Plan in U.K.

5. Shri A. K. Lahiri, J.S.O., was deputed for training in the field of applied research on corrosion under the Colombo Plan in U.K.

6. Shri R. K. Dubey, J.S.O., was deputed for training in the field of operation and control of electric arc furnace and allied furnaces used for production of alloy steel under the Colombo Plan in U.K.

7. Shri R. A. Sharma, S.S.O., was deputed for training in the field of extractive metallurgy under the Colombo Plan in U.K.

### NOMINATIONS

Dr B. R. Nijhawan, Director, National Metallurgical Laboratory, has been re-elected for a second term in succession as (a) President of the Institute of Indian Foundrymen, (b) Vice-Chairman of the Structural and Metals Division Council (SMDC) of the I.S.I.

Dr. B. R. Nijhawan, Director, has been nominated:

1. To serve as a Member of the re-constituted Metallurgy Advisory Committee of the Dept. of Atomic Energy, Government of India.
2. To serve as a Member of the Engineering Division Council of the Indian Standards Institution.
3. To serve as a Member of the Committee constituted by the Ministry of Steel, Mines & Fuel (Dept. of Iron & Steel), New Delhi, to examine the material already available on Salem ore and Neyveli lignite and to report on the suitability of these for making iron.

Dr. T. Banerjee, Dy. Director, has been nominated:

1. To serve as a Member on the Editorial Board of the *Journal of Electroplating* of Messrs Graner and Weil (India) Ltd., Bombay.
2. As a Member and Convener of the Anodes for Electroplating Subcommittee, ETDC 12:5 of the I.S.I.

Dr. E. G. Ramachandran, Asst. Director, has been nominated:

1. To serve as a Member of the Development Council for Non-ferrous Metals & Alloys of Govt. of India.
2. To serve as a Member of Copper & Copper Alloys Sectional Committee, SMDC 11 of the I.S.I.

Shri R. Choubey, S.S.O., has been nominated to serve as a Member on the "Methods of Non-destructive Testing" Subcommittee SMDC 3-1 of the I.S.I.

Shri Jatinder Mohan, S.S.O., has been nominated:

1. To serve as a Member on the Coke Subcommittee CDC 14:5 of the I.S.I. for a period of 3 years.
2. To serve as a Convener of the Panel for Glossary of Terms Relating to Foundry Technology SMDC 1/P-5 of the I.S.I.
3. As Convener and Member of the Panel for Coke Consumed in Cupolas (Foundries), CDC 14:5:2 of the I.S.I.

## DISTINGUISHED VISITORS

During the year under review, the following distinguished visitors visited the N.M.L.

1. His Excellency MR. TAGE ERLANDER, Prime Minister, Sweden, and Mrs. Erlander
2. MR. ASTRON, Head of the Political Dept., Ministry of Foreign Affairs, Sweden
3. MR. SHEREMOLIEV, Member of the Council of Foreign Economic Relations of the Council of Ministers, U.S.S.R.
4. DR. A. G. WHITE, team leader of the experts under the United Nations Technical Assistance Programme for investigation of indigenous substitutes of imported raw materials and utilization of existing production facilities in the country
5. MR. HUGH B. RIPMAN, of International Bank for reconstruction and development
6. MR. R. A. BEIJER, General Secretary, International Association for the Exchange of Students for Technical Experience
7. DR. JAMES GREIG, a delegate from British Association for the Advancement of Science
8. MR. K. S. BLASKETT, Principal Research Officer from the University of Melbourne, Australia
9. SIR EWART SMITH, delegate from the Royal Society, U.K.
10. An eight-member German delegation of educationists from the German Democratic Republic (East Germany), headed by Dr. Wilhelm Girus, State Secretary for Special and Higher Learning
11. Members of American Steel delegation
12. DR. H. J. BHABHA, F.R.S., Chairman of the Indian Atomic Energy Commission, accompanied by the Members of U.S.A & U.S.S.R. Atomic Energy Commission.
13. MR. M. DARGENT, Member of French Atomic Energy Commission.
14. MR. JAMES J. BLAKE, American Consul-General, Calcutta
15. MR. S. M. YUSUF, Director-General of Mineral Resources, Govt. of Pakistan
16. Members of the Estimates Committee (Eastern Zone) under the chairmanship of Mr. H. C. Dasappa
17. MR. P. C. NEOGI, Director-General, Research, Design & Standardization Organization, Ministry of Railways, New Delhi
18. MR. T. N. SINGH, Member, Planning Commission, Industries, Govt. of India
19. MR. G. L. BANSAL, Secretary-General of Federation of Indian Chamber of Commerce and Industry, New Delhi
20. MR. KASTURBHAI LALBHAI, Chairman, National Research Development Corporation, New Delhi
21. GEN. K. S. THIMAYYA, Chief of Army Staff, Govt. of India
22. LT-GEN. K. BAHADUR SINGH, Commandant, National Defence College, Ministry of Defence, Govt. of India
23. MR. A. SRINIVASAN, Dy. General Manager of Neyveli Lignite Project
24. MR. P. H. HARIHARAN, Financial Editor of the *Times of India*, Bombay
25. MR. KODANDARAMAN, I.C.S., Commissioner of Chotanagpur



## GENERAL

During the year under review four pilot plants relating to (i) Beneficiation of low-grade Indian manganese, chrome and other ores by ore-dressing and thermal beneficiation techniques, (ii) Production of electrolytic manganese and manganese dioxide from low-grade Indian manganese ores, (iii) Aluminizing of steel wire and sheets, and (iv) Production of Refractories have reached the final stages of completion with the necessary equipment and machinery fabricated from indigenous resources based essentially on the design worked out by the laboratory's own technical staff. The pilot low-shaft furnace has been under operation round the clock, producing commercial grades of foundry pig iron from non-coking coal. Steady progress was maintained on all other research projects under way.

The proceedings of the Symposium on "Iron and Steel Industry in India" were printed and circulated to all the authors who contributed for the Symposium and to scientific organizations for review purposes. As much as 127 technical enquiries were attended to and 12 *ad hoc* investigations were undertaken on behalf of the industries and Government organizations. The Symposium on "Pilot Plants in Metallurgical Research and Development" organized by the National Metallurgical Laboratory from 15 to 18 February 1960 was attended by eminent scientists from all over the world and short proceedings of the Symposium was also brought out for circulation in the technical journals in India and abroad. Four patents were filed, two patents were sealed and five patents were accepted.

# APPENDIX I

## PAPERS PUBLISHED

Proceedings of the Symposium on "Iron and Steel Industry in India", organized by the National Metallurgical Laboratory containing all the papers presented, were published during the period under review: Four issues of the *NML Technical Journal* were brought out during the year under review. Besides the above, the following papers and articles were published in leading Indian and foreign journals.

### Research Papers

1. VED PRAKASH & ENTWISTLE, K. M., "The Effect of Specimen Size on the Quench Ageing of an Aluminium 3.8 per cent Copper Alloy", *J. Inst. Metals (London)*, **87** (1959), 262-264.
2. RAO, M. R. K., SEN, P. C. & BHASKAR RAO, H. V., "Mineralogical and Refractory Properties of Almorah Magnesite" *Refractories J.*, **9** (1959), 270, 271; and *Trans. Indian Cer. Soc.*, **17** (1958), 67-71.
3. MISRA, R. N., SEN, M. C. & BHATNAGAR, P. P., "Thermal Beneficiation of Low-grade Chrome Ores Part I — Gaseous Reduction", *Trans Indian. Inst. Metals*, **12** (1959), 221-234.
4. BANERJEE, T., "Structure of Electrolytic Alloys", *NML Tech. J.*, **1**(2) (1959), 15-22.
5. BANERJEE, S. K. & NARAYANAN, P. I. A., "Processing of Magnetite Ores with Particular Reference to Salem Magnetite", *NML Tech. J.*, **1**(2) (1959), 23-27.
6. MINHAS, G. S. & BHASKAR RAO, H. V., "Evaluation of Quartzite for the Manufacture of Silica Bricks", *NML Tech. J.*, **1**(2) (1959), 32-37.
7. LAHIRI, A. K. & BANERJEE, T., "Cavitation Erosion — A Case of Conjoint Action", *NML Tech. J.*, **1**(2) (1959), 38-40.
8. ARORA, S. M., GUPTA, P. K. & NIJHAWAN, B. R., "Linseed Oil as An Organic Flux in Hot-dip Aluminizing of Steels", *NML Tech. J.*, **1**(3) (1959), 9-16.
9. BANERJEE, N. G., "Vacuum Determination of Hydrogen in Steel", *NML Tech. J.*, **1**(3) (1959), 17-21.
10. GUPTA, M. K. & KAR, B. C., "Spectro-chemical Analysis of Aluminium Alloys by the Oxide Arc Technique", *NML Tech. J.*, **1**(3) (1959), 23-27.
11. RAO, M. R. K., SEN, P. C. & BHASKAR RAO, H. V., "Magnesite Crucibles for Use in High Frequency Induction Furnace", *NML Tech. J.*, **1**(3) (1959), 28-32.
12. SRINIVASAN, S. R., GURDIAL SINGH & KAR, B. C., "Battery Active Manganese Dioxide from Low-grade Manganese Ores by Chemical Process", *NML Tech. J.*, **1**(3) (1959), 33-36.

13. BHASKAR RAO, H. V., "A Study of Sheavaroy Bauxite Ores for the Development of High Alumina Refractories: Part I—Factors Affecting the Preparation of Grog", *J. sci. industr. Res.*, **18A** (1959), 557-563.
14. RAMACHANDRAN, E. G. & VED PRAKASH, "An Investigation on the Magnetic Properties of Some Permanent Magnet Materials", *Trans. Indian Inst. Metals*, **12** (1959), 333-348.
15. RAMACHANDRAN, E. G. & VED PRAKASH, "The Electrical and Magnetic Properties of Some Low-manganese and Low-aluminium Steels", *Trans. Indian Inst. Metals*, **12** (1959), 349-358.
16. NIJHAWAN, B. R., "The Ageing of Steel", *Blast Furnace and Steel Plant*, **5** (1959) and *Tisco Tech. J.*, **5** (1958), 177-182.

### General Technical Papers

1. NIJHAWAN, B. R. & SEN, B. L., "Alternative Methods of Iron Making", *Statesman Special Supplement on Indian Coal*, 31 July 1959.
2. TREHAN, Y. N., "The Structure of Sulphide Layers on Copper", *Trans. Indian Inst. Metals*, **12** (1959), 387-395.
3. BHATNAGAR, P. P. & NIJHAWAN, B. R., "The Role of N.M.L. in the Development of Ferro-Alloy Industry in India", *Trans. Indian Inst. Metals*, **12** (1959), 257-271.
4. NIJHAWAN, B. R., "Non-ferrous Metal Industry in India — Excellent Prospects", *Major Industries of India Annual*, **8** (1958-59), 145-157.
5. BHASKAR RAO, H. V. & NIJHAWAN, B. R., "Recent Trends in Steel Plant Refractory Practice", *Proc. of Defence Metallurgical Conference* (103-104), held from 1 to 5 December 1958.
6. BANERJEE, T., "Metallurgical Research in India", *Proc. of Defence Metallurgical Conference*, (105-112), held from 1 to 5 December 1958.
7. NIJHAWAN, B. R., "A Survey of Developments in Foundry Technology and Indian Foundry, Industry", *Iron & Steel Review*, **II** (1959), 1137-1144.
8. NIJHAWAN, B. R., "The Role of N.M.L. in Metallurgical Research and Development in India", *Proc. of Symp. on Iron and Steel Industry in India* (62-71).
9. CHATTERJEE, A. B. & NIJHAWAN, B. R., "Low-shaft Furnace Smelting of Pig Iron in India", *Proc. of the Symp. on Iron and Steel Industry in India* (244-251).
10. CHATTERJEE, A. B., "Technological Aspects of Low-shaft Furnace Process", *Proc. of Symp. on Iron and Steel Industry in India* (252-258).
11. NIJHAWAN, B. R., "Recent Trends in Iron and Steel Technology", *Proc. of the Symp. on Iron and Steel Industry in India* (292-307); and *Blast Furnace & Steel Plant*, October 1959.
12. NIJHAWAN, B. R., "Iron & Steel Industry in India", *Proc. of the Symp. on Iron and Steel Industry in India* (322-336).
13. KRISHNAN, R. M., SRIVASTAVA, K. N. & BANERJEE, T., "The Development of Iron and Steel Industry in India's Five Year Plans", *Proc. of the Symp. on Iron and Steel Industry in India* (342-352).



14. RAMACHANDRAN, E. G. & BALASUNDARAM, L. J., "The Nature and Structure of the Sigma Phase in Transition Alloys", *NML Tech. J.*, **1**(2) (1959), 28-31.
15. SEN, B. L., CHATTERJEA, A. B. & NIJHAWAN, B. R., "Refining of Indian Pig Iron in an Experimental L-D Oxygen Converter: Part I", *Proc. of the Symp. on Iron and Steel Industry in India* (192-199).
16. SEN, B. L., CHATTERJEA, A. B. & NIJHAWAN, B. R., "Refining of Indian Pig Iron in an Experimental L-D Oxygen Converter: Part II" *Proc. of the Symp. on Iron and Steel Industry in India* (200-210).
17. KRISHNAN, R. M., "Foundry Industry in Great Britain and General Trends of Development", *NML Tech. J.*, **1**(4) (1959), 5-15.
18. KAPOOR, A. N., "Some Recent Developments in Protective Coatings in U.K.", *NML Tech. J.*, **1**(4) (1959), 16, 17.
19. BANERJEE, N. G., "Determination of Gases in Metals and Alloys by Vacuum Technique", *NML Tech. J.*, **1**(4) (1959), 28-33.
20. DAS, B. N., "Some Recent Developments in Metal Working Techniques", *NML Tech. J.* **1**(4), (1959), 34-40.
21. NIJHAWAN, B. R., "Scope of Pilot Plant Research and Development", *NML Tech. J.*, **2**(1) (1960), 11-23.
22. VED PRAKASH, "Wire Drawing", *Tisco Tech. J.*, **6**(4) (1959), 215-223.
23. BHATNAGAR, S. S. & GUPTA, P. K., "Extrusion of Metals", *Tisco Tech. J.*, **6**(4) (1959), 224-233.
24. DAS, B. N., "Surface Finishing and Cladding of Metals: Part I — Surface Finishing of Metals", *Tisco Tech. J.*, **6**(4) (1959), 260-266.
25. DAS, B. N., "Surface Finishing and Cladding of Metals: Part II — Cladding of Metals", *Tisco Tech. J.*, **6**(4) (1959), 267-271.
26. RAMACHANDRAN, E. G. & MAJI, K. D., "Cold Working and Recovery Textures", *Tisco Tech. J.*, **6**(4) (1959), 272-279.
27. CHATTERJEA, A. B., "Measurement of Internal Stresses in Metals and Alloys", *Tisco Tech. J.*, **6**(4) (1959), 280-289.
28. NIJHAWAN, B. R., "Special Metal-working Techniques", *Tisco Tech. J.*, **6**(4) (1959), 290-298.
29. MATHUR, G. P. & NARAYANAN, P. I. A., "Beneficiation and Processing of Some Important Raw Materials for Iron and Steel Production, *Proc. of Symp. on Iron and Steel Industry in India*" (136-142).
30. "Pilot Plant Developments: Part III — Low-shaft Furnace Pilot Plant at the N.M.L., Jamshedpur, *Research & Industry*", **4**(4) (1959), 81-84.
31. "Accelerated Fatigue Testing of Steel", *Research & Industry*, **4**(4) (1959), 133, 134.

The following articles were published in the special issue of *Statesman Supplement*, 15th February 1960, entitled, "Pilot Plants in Metallurgical Research and Development", issued on the occasion of the inauguration of the Symposium on this subject, organized by the N.M.L.:

1. NIJHAWAN, B. R., "Scope for Pilot Plant Research and Development at the N.M.L".
2. DHANANJAYAN, N., CHAKRABARTI, H. K. & BANERJEE, T., "Production of Manganese and Manganese Dioxide".

3. PRASAD, T. V. & BHASKAR RAO, H. V., "Metallurgical Refractories with Indian Raw Materials".
4. KAPOOR, A. N., GUPTA, P. K. & NIJHAWAN, B. R., "Hot-dip Aluminizing of Steel Wire".
5. CHATTERJEA, A. B., "Pilot Plants in Pyrometallurgy".

### Letters to the Editor

1. RAMACHANDRAN, E. G. & DASARATHY, C., "Austenite Transformation and the Greninger-Troiano Technique", *J. Iron & Steel Inst.*, **192** (1959), 268.

### Papers Presented at Symposia and Technical Seminars

The following papers were presented at the Symposium on "Pilot Plants in Metallurgical Research and Development", held under the auspices of this laboratory from 15 to 18th February 1960:

1. Scope of Pilot Plant Research and Development at the N.M.L.—B. R. Nijhawan
2. Metallurgical Pilot Plants—Theory and Practice—M. J. Shahani & T. Banerjee
3. Some Aspects of Pilot Plant Research and Development in Iron and Steel Industry—B. R. Nijhawan
4. Pilot Plants in Pyrometallurgy—A. B. Chatterjea
5. Recovery of Vanadium Pentoxide from Vanadium Bearing Titaniferous Magnetites—B. K. Agrawal, M. P. Menon & P. P. Bhatnagar
6. Hot-dip Aluminizing of Steel Wire—Laboratory Scale Investigations, Pilot Plant Studies—A. N. Kapoor, P. K. Gupta & B. R. Nijhawan
7. Development of Metallurgical Refractories with Indian Raw Materials—Pilot Plant Studies—T. V. Prasad & H. V. Bhaskar Rao
8. Production of Manganese and Manganese Dioxide—Pilot Plant Investigations—N. Dhananjayan, H. K. Chakrabarti & T. Banerjee

The following papers were presented at the Symposium on "Iron and Manganese Ores", held at Calcutta in March 1960 under the auspices of Geological, Mining and Metallurgical Institute:

1. Beneficiation of Low-grade Manganese Ores of India—G. V. Subramanya & P. I. A. Narayanan
2. Beneficiation and Agglomeration of Iron Ores—S. K. Banerjee, P. V. Raman & P. I. A. Narayanan
3. Flotation of Manganese Ores—B. L. Sen Gupta, G. V. Subramanya & P. I. A. Narayanan

The following papers were presented at the Symposium on "Beneficiation of Minerals" at the Indian Institute of Science, Bangalore, held in September 1959:

1. Amenability of Low-grade Manganese Ores of Mysore to Beneficiation by Ore-dressing Methods — S. B. Das Gupta & P. I. A. Narayanan
2. Selective Flotation of Zircon from Beach Sands — G. V. Subramanya
3. Beneficiation of Ores by Chlorination — R. A. Sharma & P. P. Bhatnagar

The following papers were presented at the Calicut Session of the Indian Ceramic Society, held in February 1960:

1. Studies on the Development of Zircon Refractories from Travancore Beach Sand — T. V. Prasad, M. C. Kundra & H. V. Bhaskar Rao
2. Studies on Indian Refractory Clays: Part III — Some Clays from South India — T. V. Prasad & H. P. S. Murthy

The following paper was presented at the Annual Session of the Indian Ceramic Society in April 1959 at Kanpur:

1. Development of Graphite Crucibles from Indian Raw Materials — H. P. S. Murthy & T. V. Prasad



## APPENDIX II

### INVESTIGATION AND RESEARCH REPORTS PREPARED DURING THE PERIOD UNDER REVIEW

1. Beneficiation of Manganese Ore from Rejects after Hand Picking from S.G.B.K. Mines, Gurda, Keonjhar, Orissa (IR 155/59)
2. Semi-pilot Plant Studies on Beneficiation of Ferruginous Manganese Ores Employing the Patented Process (51625), (IR 156/59)
3. Studies on the Beneficiation of Low-grade Manganese Ore from Kurro-Madai Mines, Jabalpur, M.P. (IR 157/59)
4. Beneficiation of Low-grade Manganese Ores with Particular Reference to Semi-pilot Plant Studies on a Low Temperature Magnetizing Reduction Process for Ferruginous Manganese Ores (IR 158/59)
5. Beneficiation of Low-grade Ferruginous Manganese Ores (No. 2) from S.G.B.K. Mines, Keonjhar Dt., Orissa (IR 159/59)
6. Beneficiation of Low-grade Ferruginous Manganese Ore from Chitaldrug, Mysore (IR 160/59)
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